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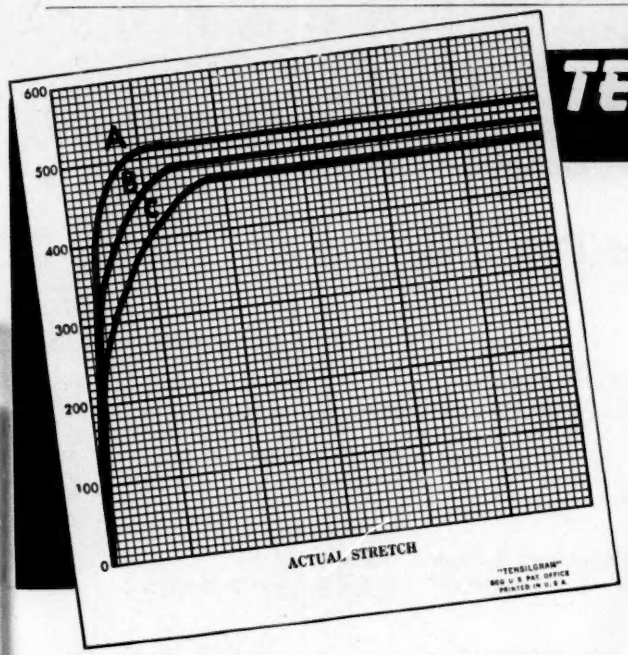

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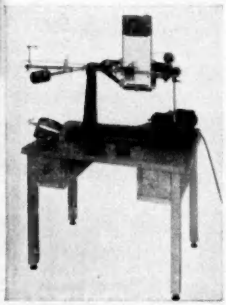

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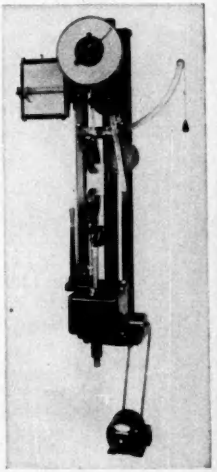

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TEXTILE RESEARCH JOURNAL

VOL. XXI, No. 5 May, 1951

The Spinning Value of Raw Cotton*

W. E. Morton†

Review of the Present Position

For some 30 years now, research workers in most of the important textile countries of the world have expended a great deal of effort in endeavors to explain the spinning performance of a cotton in terms of its measurable fiber properties. It is unnecessary to elaborate on the reasons why this problem, in addition to being of intense academic interest, is of the greatest practical importance to the cotton breeder.

In nearly all of the work done so far, the procedure has been to carry out spinning tests on a large number of cottons; make laboratory measurements on samples of the fibers; and then, by statistical processes of simple, partial, and multiple correlations, to express some chosen criterion of spinning performance in terms of such measured fiber properties as are found to have any bearing on the issue.

The outcome is a "prediction formula," taking the form of a linear equation:

$$Q = aL + bF + cS + dM + \cdots,$$

where Q is some chosen criterion of spinning performance; L, F, S, M, etc., are relevant measured fiber properties, such as length, fineness, strength, and maturity; and a, b, c, d, etc., are constants derived from the statistical analysis.

Two criteria of spinning performance have been used.

In India and the United Kingdom the preference has been for what is called the "highest standard warp count," H.S.W.C. This is the count for a warp spun yarn beyond which the lea breaking length, or lea count × strength, falls below some arbitrarily chosen, minimum acceptance level. (See Figure 1.)

For any given cotton, and within the range of counts normally spun, lea breaking length decreases approximately linearly as the count increases. The procedure, therefore, is to spin each sample with warp twist, to a comparatively narrow range of the 2 or 3 counts which are expected to straddle the H.S.W.C.; plot lea breaking length against count; and note where the lea breaking length falls to the

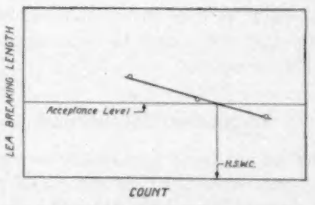

FIG. 1. *Determination of the "highest standard warp count."*

* Paper presented at the Annual Meeting of Textile Research Institute, New York City, Nov. 16, 1950.

† Professor of Textile Technology, College of Technology, University of Manchester, England.

acceptance level. If necessary, extrapolation has to be resorted to.

In the prediction formula, then, Q is the H.S.W.C.

In Egypt and the U. S., on the other hand, the criterion adopted has usually been the lea strength or lea breaking length of the cotton when spun to some arbitrarily chosen count—*i.e.*, Q is the lea breaking length or lea strength. The weakness here lies in the fact that either the investigation must be restricted to a narrow range of cottons for which the chosen count is suitable—which has obvious experimental objections—or many of the samples must be judged on their performance when spun to a count for which they would never ordinarily be used in industrial practice.

Webb and Richardson [11] have attempted to avoid these limitations and objections by spinning each cotton into a range of 3 counts chosen in reference to its staple and treating yarn count as one of the independent variables. This results in a count term, mC, being included among the fiber-property terms on the right-hand side of the equation

$$Q = aL + bF + cS + dM + \cdots + mC.$$

Such a step undoubtedly leads to a prediction formula for strength that is of greater practical use than those previously obtained by the same general method of investigation. On the other hand, it does nothing to advance our understanding—indeed, it tends to obscure the issue—of how fiber properties determine spinning behavior.

The reason for this is that the sensitivity of a cotton to changes in count is itself a function of at least one fiber property—namely, fineness. Hence, the constant m in the count term is not really a constant at all, and, strictly speaking, can only be applied with accuracy to a cotton having a value for fineness that is somewhere near the mean fineness of the samples used in deriving the equation. The importance of what is involved here is greatly accentuated when we note that Webb and Richardson [11] found this count term to be of greater weight than any of the others.

Anomalies Encountered

It would be too harsh a judgment to say that the above methods have been useless. The prediction formulas derived have been tolerably accurate for application to cottons very similar to those used for finding the formulas. On the other hand, the general outcome of the work has been to raise more questions than have been answered, and, altogether, the situation as it now stands is very far from being intellectually satisfying. The mere posing of the questions will be enough to indicate the anomalies, the contradictions, and the confusion of the situation at which we have now arrived.

It is to be expected that there should be quantitative discrepancies between the results of different workers, if only because of differences in the methods of measurement employed, of both fibers and yarns, as well as in the details of spinning. These are of interest, but of minor importance. What really merit our attention are inconsistencies of a much more fundamental kind.

Below are a few of the more important questions:

Why is it that when the same investigator, using a consistent technique, divides his entire population of samples into one or more groups he arrives at different—and sometimes markedly different—prediction formulas for the different groups?

Why is it that when correlations between actual and calculated spinning performance exceed 0.9 by a comfortable margin outstanding anomalies are nevertheless noted? Turner and Venkateraman, for example [9], found that 1 in 5 answers for Indian cottons were wrong by more than 20%.

Why is it that Turner [9], Underwood [10], and Morton [5], to mention a few, have all found that fiber strength apparently plays a completely insignificant part in determining yarn strength? Even making allowance for the fact that in these cases fiber strength was measured over 1-cm. lengths, this particular result cannot be accepted as true.

Why is it that length is found to be more important than fineness for Indian cottons [9], whereas the reverse is found to be the case for other cottons?

Why is it that Gulati and Ahmad [2] find that for Indian cottons over 0.77 in. in length, high fiber maturity is advantageous, whereas for shorter cottons, the reverse is true?

Why does Underwood [10] find that length is more important for Empire cottons than for Egyptian cottons?

And, finally, why do Webb and Richardson [11] find that for American cottons shorter than 1 in., fineness is the most important fiber character, whereas for those 1 in. and longer, strength is the most important?

The Need for a New Approach

Attempts have been made to answer some of these questions, notably by Turner and Venkateraman [9], but in the author's opinion no really convincing answer has yet been given to any of them. The reason is that we have been attempting the impossible. As already concluded by Balls [1] over 20 years ago, the yarn strength obtainable from any cotton cannot be expressed in terms of fiber properties—rationally, at any rate—by using a single linear equation.

What is called for is a complete re-examination of what is involved in the problem, and the devising of an entirely new method of attack.

This has been done for us by Spencer-Smith [6] of the Linen Industries Research Association. Although primarily concerned with linen yarns, he has treated the subject as a fundamental textile problem, without distinction between one fiber and another.

A clue to the explanations for at least some of the inconsistencies we have noted may be seen if we consider the "spinning-test line"—*i.e.*, the plot of breaking length against count (Figure 2). It is evident that the point P, representing a particular value of H.S.W.C.—or strength at a given count—, could, in practice, be on any number of spinning-test lines. It is further evident from the figure that the physical properties of a cotton giving rise to line A, for example, must be quite different from those of a cotton giving rise to line B. The relation between spinning performance and fiber properties, therefore, cannot be linear.

What we want is a prediction formula that will locate not a point, but a line.

The realization of this need focuses attention on the shape of the line and on the nature and causes of the increasing yarn irregularity which is responsible for the falling-off of strength as count increases.

In this connection, an entirely new line of thought was opened up as a result of the studies of Spencer-Smith and Todd [7] of the L.I.R.A., and Martindale [3, 4] and Daniels [1a] of the Wool Industries Research Association. Both of these parties arrived independently at the same conclusion—namely, that the underlying irregularities of any yarn are random in origin and are therefore amenable to the application of statistical theory.

If these were the only irregularities, the coefficient of variation of weight per unit length of yarn, V_R, would be given by

$$V_R^2 = \frac{(100)^2}{N} + \frac{V_m^2}{N},$$

where V_m is the coefficient of variation of fiber weight per unit length; and N is the average number of fibers in a cross section of the strand.

In yarns, the "statistical" irregularity is supplemented by irregularities arising in various ways from the processing machines. But Spencer-Smith put forward the hypothesis that for all yarns spun on a given system from a given raw material, the ratio of the two irregularities is constant.

In that case, since for a given raw material V_m is constant, the expression can be written

$$V_R = \frac{\text{constant}}{\sqrt{N}}.$$

Proceeding from this point, Spencer-Smith then showed on theoretical grounds that a rectilinear spinning-test line should be obtained if single-thread breaking length (not lea breaking length) is plotted against the square root of the count (Figure 3).

Experimental verification of his theory and justification of his hypothesis were provided by the results of numerous spinning tests on raw flax. When single-thread breaking length was plotted against count, straight lines were, in fact, obtained. Similar support in the case of worsted yarns was found from some of the data published by Stanbury and Byerley

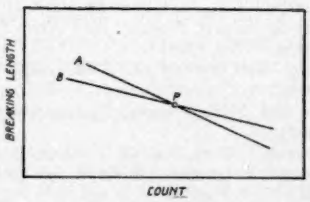

FIG. 2. *Plot of breaking length against count.*

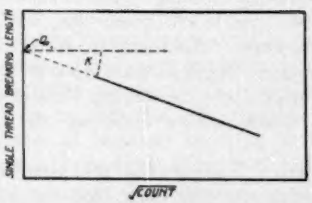

FIG. 3. *Determination of* Q_0 *and* K *from a spinning-test line.*

in 1934 [8]. Recent, and as yet unpublished work by Dr. Nanjundayya and the author has shown that a straight line is also obtained for $1\frac{7}{8}$-in. Fibro when spun on the cotton system. A test of the theory by the spinning of cotton yarns remains to be made.

Two Separate Prediction Formulas Required

Let us consider what it means if this linear relationship is generally true for all yarns, and, in particular, for cotton yarns. It means that the equation for the spinning-test line based on single-thread tests is

$$Q_c = Q_o(1 - K\sqrt{C}).$$

In order to determine Q_c, the product of single-thread count and strength at any count, C, two quantities are required—Q_o and K—both of which may be determined by a suitably conducted spinning test. It then remains to find prediction formulas for these quantities in terms of the relevant fiber properties.

K is the slope, indicating the rate at which single-thread breaking length falls off as the count spun becomes finer. From Spencer-Smith's theory, this is largely dependent upon the average number of fibers per unit cross section of the yarn—*i.e.*, fiber fineness. Experimental support for this is to be found in some of the present author's earlier work [5]. Thus, in spinning trials on a range of cottons in 1926, the coefficient of correlation between hair weight per cm. and the fall in lea breaking length between 24's and 40's was found to be 0.92, which doesn't leave a great deal of room for other fiber properties to play any notable part. Theory suggests, however, that fiber length, at least, must also be taken into consideration; while fiber maturity, neglected in Spencer-Smith's analysis, may indirectly affect the behavior of the cotton during the drafting operations.

Q_o is the intercept of the extrapolated spinning-test line on the OY axis (Figure 3), and represents the single-thread breaking length of the cotton concerned when spun to zero count—*i.e.*, into a yarn of infinite thickness. Alternatively, it is the single-thread breaking length of what Spencer-Smith calls the "fracture zone"—the shortest element of a yarn specimen which wholly embraces the region of rupture.

With either interpretation the yarn-irregularity factor vanishes at this point, and the dominating fiber property becomes that of fiber strength. Here again, however, other fiber properties cannot be neglected. Thus, length will affect the incidence of end-slip in the fracture zone; while fineness and surface characteristics will affect fiber cohesion under the influence of twist. In addition, according to strict theory, the interfiber variation in extension characteristics should also be taken into account; but because of the comparatively disorderly arrangement of fibers in a yarn, it seems unlikely that the influence of this particular factor will be detected. Only experiment can show whether this is so or not.

Unfortunately, neither K nor Q_o bears a simple linear relation to the fiber properties upon which it depends. Consequently, the application of straightforward correlation methods in the determination of these relationships could be criticized on the same grounds as mentioned earlier. On the other hand, it would be reasonable to use this simple method of attack in the first instance in order to find out how much error is involved, because it is possible that some of the complicating factors indicated by theory are of negligible importance. Again, only experiment can show.

Although we can now see the nature of the problem more clearly, it is evident that it is one by no means easily solved. It may be that a completely rigorous solution is beyond reach; but an experimental study of fiber properties in relation to the two quantities Q_o and K, instead of to only one arbitrarily chosen criterion of yarn strength, should take us nearer to the goal.

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(*Manuscript received December 18, 1950.*)

Mechanics of Elastic Performance of Textile Materials

Part VII: Mechanical Properties of Hard Fibers with Reference to Their Use in Cordage Structures*

Ernest R. Kaswell and Milton M. Platt

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Abstract

As a first step in studying the factors which determine the efficiency of cordage structures, a series of related experiments have been performed on the common cordage fibers. The basic mechanical properties of Manila and domestic Abaca, Sisal, Henequen, and Sansevieria fibers have been determined by a study of their one-time loading to rupture and repeated-tension characteristics. Data are presented on: (1) those fiber properties deemed most significant in terms of cordage use; (2) the effects of processing fibers into yarns; and (3) mechanical properties under varying ambient atmospheric conditions representing some of the extreme environments under which cordage is used.

THE CORDAGE INDUSTRY, similar to other parts of the textile industry, is in the somewhat paradoxical situation of being one of the older fields of endeavor of man, but yet having the least background of science and technology at its disposal. While this lack of ordered knowledge may not have prevented the empirical development of natural materials to their best advantage by continued trial and error procedures over the years, the last world conflict proved the inadequacy resulting from such a development from both an economic and a national-security point of view.

The outbreak of hostilities in the Philippines, our major source of Abaca fiber, created a critical shortage of this valuable material. There was available neither time, material, nor personnel to investigate whether the supplies on hand were being used to their best advantage from a construction point of view, nor had any work been done to show that the fiber could be stock-piled and stored for any extended period of time without loss of essential characteristics. Only in one case [9] had the basic mechanical properties of a group of hard fibers been examined; and extensive work had not been done on possible synthetic substitutes such as nylon.

Following the cessation of hostilities it became obvious that the following steps should be taken in order to both obviate the technical difficulties in case of a recurrence of the critical cordage deficiency and ensure a sufficient technological know-how in both a normal economy and in the event of another emergency:

(1) The fundamental mechanical properties of the cordage fibers must be known. Differences and similarities between materials must be established, and the results classified and utilized in explaining differences in the properties of cordage structures made from these materials. It will be noted from the data presented herein that the mechanical properties of the cordage fibers are such that very sensitive and precise equipment is necessary to define their tensile properties because of their extremely low elongation.

(2) The influences of construction in altering the properties of the cordage fibers must be understood, and the general principles of the effects of rope

* Part of a research program on cordage sponsored by the U. S. Navy, Office of Naval Research and Bureau of Ships.

Parts I and II of this series appeared in the February and December, 1948, issues, respectively, Part III in the January, 1950, Parts IV and V in the August, 1950, and Part VI in the October, 1950, issues.

geometry on the translation of fibers into cordage structures formulated.

With a knowledge of both the basic fiber properties and general principles of translation, it is possible to visualize the efficient design of cordage materials for various uses. This "applied mechanics" of rope structures represents the long-range aspect of the research now under way, this paper presenting data on the mechanical properties of hard fibers deemed most significant in terms of cordage performance.

Mechanical Properties of Hard Fibers

The term "hard fiber," as used in this work, refers to the fiber of commerce. Practically all of the cordage fibers are actually bundles of individual cell fibers, cemented to each other along both their sides and ends to form essentially a ribbon. The reader is referred to the excellent text by Weindling [10] in which is given a discussion of the basic morphology, chemistry, origin, and methods of processing of the various cordage materials.

A—Selection of the Test Method and Test Conditions

There are a multitude of simulated service tests which have been developed for the purpose of comparing the performance in use of cordage structures. From a practical standpoint, such tests often provide useful information on the comparative durability of various materials and constructions made therefrom. However, it has recently been recognized that of much greater importance, from the long-range point of view, is a knowledge of the basic properties of materials, developed through simple, fundamental-type tests which are aimed at defining these basic properties and explaining the performance of materials in terms of such qualities. The work of Berkley [2] on orientation and fiber strength, of Hamburger [3] on abrasion and energy absorption, and of Meredith [7], to mention a few, are representative of the value of this approach for textiles.

Undoubtedly, with respect to the basic properties of textile materials, the simple tensile test yields the most informative fundamental knowledge. This is so for the following reasons: (1) Being flexible materials, in the engineering sense, the major stresses produced in normal service are of a tensile character; (2) The visco-elastic properties of materials have been shown to be of great importance in use, and these may be conveniently studied by tensile testing [4, 6]; (3) The extremely high variability of natural textile materials, such as hard fibers, presents the necessity for testing under as controlled and sensitive conditions as possible. This can be accomplished by the use of recently developed resistance wire strain-gage tensile testers.

This study is therefore concerned with the determination of those basic properties indicated by the tensile performance of cordage materials, utilizing the Instron-type autographically recording constant-rate-of-elongation loading devices, equipped with resistance wire strain-gages for the determination of load, and with magnification of the elongation axis. All except the wet and cold room tests were made at 70°F and 65% R.H., using a 10-in. sample gage length and a 3 in. per mi. rate of elongation.

B—Mechanical Properties of Hard Fibers

Manila Abaca.—Manila Abaca cordage represents the standard of performance required in ropes of excellent quality. The major initial portion of this research is devoted to a detailed study of this material. Pedigreed lots of this fiber actually consisting of various grades ("grades" refers to classification within a fiber type based on location of the fiber within the plant) have been studied.

TABLE I. TENSILE PROPERTIES TO RUPTURE * OF UNOILED MANILA ABACA FIBER

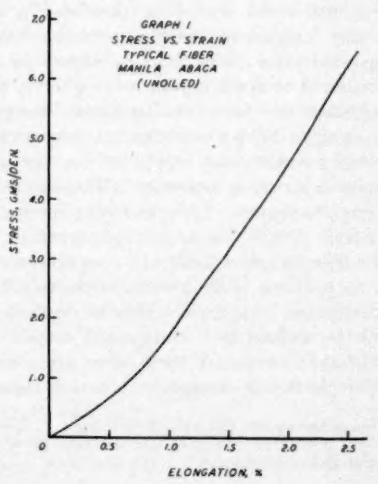
Fiber grade	Fiber denier	Tensile strength (lbs.)	Tenacity (g./den.)	Rupture elongation (%)	Energy absorption (g. cm./cm. den.)
F	288	4.88 (33.2)†	7.58 (18.3)	2.60 (21.5)	86×10^{-3}
G	444	6.73 (22.2)	7.00 (13.3)	2.99 (18.7)	68×10^{-3}
I	450	6.44 (33.0)	6.58 (24.5)	2.74 (23.1)	64×10^{-3}
J-1	377	5.33 (33.2)	6.35 (25.0)	2.48 (24.0)	74×10^{-3}
J-2	373	4.90 (27.6)	6.07 (20.5)	2.75 (18.6)	100×10^{-3}
Average‡	380	5.60 (30)	6.71 (21)	2.65 (22)	74×10^{-3} (19)

* All values are averages of 25 tests except those for J-1, for which 75 breaks were made.
† Figures in parentheses are coefficients of variation (%).
‡ Average weighted for grade distribution: F, 10%; G, 30%; I, 30%; J-1, 20%; J-2, 10%.

In Table I are given the tensile properties of the various grades of the Manila Abaca fiber. In addition to the average values given for each grade of fiber, a grand average is also given for "Abaca fiber" per se. This average is determined by weighting the individual averages of each grade according to their distribution within an "oiled and combed" roving, which is the cordage product that is ultimately twisted into yarn. This weighted averaging enables the determination of the effects of oiling and combing. For this data, as well as for all other data reported herein, each fiber was weighed prior to testing so that precise control of unit properties (tenacity, etc.) could be maintained.

The calculation of the energy absorption should be described. This quantity represents the area to rupture under the stress-strain curve. To avoid the planimetering of a tremendous number of individual curves, an average curve for each grade was constructed by arithmetically averaging the stresses at equal strains in each of the 25 breaks per grade, from zero strain to the average rupture strain. The area under this average curve is considered to be the energy absorption. Hence, the coefficient of variation for the energy absorption refers to only 5 individual test values. A typical stress-strain curve for unoiled Manila Abaca is given in Graph 1.

Examination of Table I shows all the grades to be statistically alike with respect to tenacity and elongation to rupture. The variability is seen to be great—in particular, the variability in tensile strength. The advantage of weighing each fiber prior to testing is apparent from Table I, wherein it is indicated that by so doing the rupture-strength variability is reduced from 30% to 21%. This indicates that fiber strength is functional with fiber weight, and that strength is most precisely defined on a specific weight basis. The residual variability of 21% is probably representative of the inherent variability of these natural fibers. It is also to be noted that the elongation to rupture has a variability of 22%, about the same as that of the tenacity. However, the strongest grade does not possess the greatest elongation, nor is the weakest grade of the lowest elongation.



The strength of the Manila Abacas is comparable to that of the high-tenacity synthetic fibers nylon and Fortisan. However, the elongation to rupture is extremely low compared to that of these synthetics. Even of greater difference is the shape of the stress-strain curve shown in Graph 1. Here it can be seen that the typical yield region that is present in most conventional textiles is lacking, and, instead, the shape shows a structure which appears to stiffen with continuous increase in stress or strain. One of the writers has shown [8] that this type of curve causes severe losses in strength and energy absorption when the fiber is put into yarns and other fiber aggregates. This effect will be more fully described in a subsequent paper.

Other hard fibers.—In addition to Manila Abaca, other hard fibers are used in cordage form for a variety of purposes, such as binder and baler twine, light ropes, and heavy ropes. Western Hemisphere sources exist for the Sisals, Henequens, and Sansevieria, and recently Central American sources of Abaca have been established. Pedigreed lots of such fibers of different varieties have been studied, and the results are given in Table II.

It is shown in Table II that differences exist among the various fibers with respect to their tenacities and elongations. The differences in properties between varieties of the same type of fiber are negligible, and hence all the Sisals may be grouped together, as may all the Henequens and all the Abacas.

Before comparing different types of fiber, it is of interest to compare the varieties of Abaca. It is obvious from the data given in Table II that if any difference in tenacity exists among the varieties of Abaca, the Central American Abaca group possesses slightly higher values than does the Manila Abaca.

TABLE II. TENSILE PROPERTIES * OF UNOILED HARD FIBERS

Fiber type	Fiber denier	Tenacity (g./den.)	Rupture elongation (%)
Henequen			
Mexican	435	3.37 (14)†	4.93 (20)
Tampico	478	3.26 (16)	4.25 (21)
Victoria	456	3.17 (21)	5.12 (21)
Sisal			
Haitian	355	4.40 (20)	2.77 (18)
African	322	4.15 (18)	2.77 (15)
Brazilian	316	4.74 (21)	3.01 (18)
Java	390	4.46 (23)	2.33 (19)
Abaca			
Guatemala	400	6.96 (28)	2.73 (24)
Honduras	400	7.60 (23)	2.92 (20)
Guatemala-Honduras	400	7.11 (27)	2.84 (19)
Manila	380	6.71 (21)	2.65 (22)
Sansevieria	88	4.54 (25)	2.67 (20)

* All values are averages of 125 tests except those for Manila Abaca, which are averages of 175 tests.
† Values in parentheses are coefficients of variation (%).

It is, of course, true that differences in grades of the Central American Abaca relative to the Manila Abaca could be the cause for this result. However, under the worst of circumstances, it can be stated that the tensile properties of "domestic" Abaca fiber equal those of Manila Abaca fiber. Whether or not construction and/or processing effects cause different extents of translation of these fiber properties cannot be predicted at this stage of the research. However, it can be stated that Central American Abaca appears to be of great promise as a domestic substitute for Manila Abaca. The shapes of the stress-strain curves of the Abacas are substantially alike, and hence Graph 1 may be used as typical for any of the Abacas.

For purposes of comparison of fiber types, the values given in Table II for varieties have been averaged, and the resulting properties, indicative of the characteristics of the fiber type, are tabulated in Table III.

It is clear from Table III that the basic tensile properties of the materials listed differ. With respect to tenacity, the Abacas are the strongest of the group, the Sisals and Sansevieria exhibiting 65% of the tenacity of the Abacas, while the Henequens are only 47% as strong. When elongations are considered, only Henequen differs from the other fibers. Assuming, for the moment, that these properties are translated to the same degree for each type of fiber

TABLE III. COMPARATIVE PROPERTIES OF HARD FIBERS

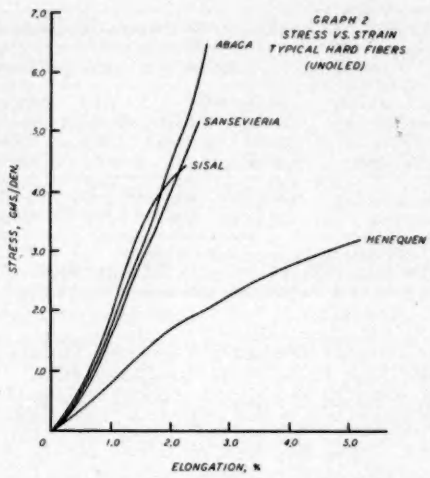
Fiber	No. of tests	Tenacity (g./den.)	Coefficient of variation (%)	Rupture elongation (%)	Coefficient of variation (%)
Abaca	550	7.0	21	2.78	21
Sisal	600	4.4	21	2.72	18
Henequen	375	3.3	20	4.77	21
Sansevieria	125	4.5	25	2.70	20

when rope constructions are considered,* and that tensile strength is the critical design factor, then it is clear that heavier rope than that for Abaca would be required for all fibers. Sisal or Sansevieria rope would require 60% more weight of fiber than would Abaca rope, while one of Henequen would require 110% more weight of fiber.

The Sisals and Henequens find great use as binder and baler twine material. It is obvious that in case of a shortage of Abaca fiber, where Sisals were directed to cordage use, Sansevieria could adequately be used to bolster the amounts of Sisal and Henequen in both binder twine and cordage use. While on a fiber-strength basis none of these materials are comparable with the Abacas, they could undoubtedly serve as temporary substitutes. It is, of course, necessary that tensile properties of higher-order structures be studied before any conclusive statements be made on this matter. Such studies represent a major part of this research.

One other factor should be considered in discussing the comparative tensile properties—that of energy absorption. This property reflects the ability of a material to resist impact loads wherein the requirement is one of converting kinetic energy into strain energy. Such a condition has to be realized in use when a sudden load is applied to a rope at, say, the start of a tugging operation. The magnitude of the energy absorption of Manila Abaca fibers is given in Table I. While comparable figures are not given for the fibers listed in Table III, comparisons can be made on the basis of the product of the tenacity and the elongation at rupture. This is justified since, as will be shown later, the general shapes of the stress-strain diagrams of these fibers are alike to a considerable degree, except for that for Henequen.

* This is known not to be the actual case. A subsequent paper will discuss the effects of inherent fiber properties on their translation.



Inspection of Table III shows that the Sisals and Sansevieria possess about 65% of the energy-absorbing ability of the Abacas, while the Henequens possess about 81%. It would then be expected that the Henequens could perform more satisfactorily than the Sisals in resisting impact loads, even though it would not be up to the standard of the Abacas. This is the case for a one-time use of the material. The energy relationships in repeated stress are discussed on p. 270.

Typical stress-strain diagrams of the Sisal, Henequen, Abaca, and Sansevieria fibers are given in Graph 2. The major difference amongst these fibers, aside from their tenacities, is the difference in stress-strain curve shapes between the Henequens and the others. Abaca, Sisal, and Sansevieria have stress-strain curves of approximately the same shape, all falling in essentially the same region. Henequen, on the other hand, possesses a curve shape that is more representative of common textile fibers, particularly in the yield region. According to the shape of the stress-strain curve Henequen should theoretically [8] produce better translation of fiber properties into cordage than the other fibers. In addition, for uses wherein high stress concentrations are known to exist, such as knotting, Henequen should perform more satisfactorily, and, as is described on p. 269, this is the case.

Wet properties of hard fibers.—While it is not the purpose of this study to investigate such effects as immersion in salt water on cordage structures, it is apparent that since Naval rope invariably becomes wet in use, some information on changes in properties is desirable. Therefore, a limited amount of work was done on the effect of immersion in a 1% aerosol solution at room temperature for 15-min. soaking time. These specimens were tested immediately after removal from the wetting solution. The results are compared with those for dry fiber in Table IV.

TABLE IV. WET PROPERTIES * OF UNOILED HARD FIBERS

Fiber type	Tenacity Dry (g./den.)	Tenacity Wet (g./den.)	Elongation to rupture Dry (%)	Elongation to rupture Wet (%)
Manila				
Abaca				
Grade F	7.58	7.03	2.60	2.60
Grade G	7.00	6.35	2.99	3.20
Grade I	6.58	7.76	2.74	3.38
Grade J-1	6.35	6.49	2.48	3.41
Grade J-2	6.07	5.31	2.75	2.64
Guatemala	6.66	8.43	2.04	2.64
Honduras	7.29	8.17	2.20	2.63
Sisal				
African	4.08	3.93	2.22	2.92
Brazilian	5.03	4.60	2.45	3.05
Haitian	4.45	4.20	2.22	2.89
Java	4.40	4.09	1.94	2.34
Henequen				
Mexican	3.21	2.78	4.00	6.23
Tampico	3.18	2.84	3.47	5.47
Victoria	3.21	2.73	4.28	6.60
Sansevieria				
Longiflora	4.63	4.07	2.43	2.72
Lorentii	4.81	4.81	2.36	2.72
Trifasciata	4.02	4.30	2.00	2.48

* Tests were made in the first half of 1948 except for those on Guatemalan and Honduras Abaca, Henequen, Sisal, and Sansevieria, which were made in the last half of 1950.

It is clear from Table IV that in the case of Manila Abaca there is no directed trend in tenacity following wetting-out. The strength of some grades is higher when wet, but for others it is lower. When the grand averages of tenacities are compared, the differences are found to be statistically insignificant. The same observation results when the effects of wetting on the elongation to rupture are considered. For all of the fibers but Henequen, statistical analysis indicates that tenacity is unaffected by wetting-out. Statistical analysis shows that for all of the fibers, the elongation is higher for wet fibers. It should be pointed out that the dry tenacities and elongations re-

ported in Table IV are not the same as those given in Table II, except for those for the Manila Abaca. The Manila Abaca wet properties were determined at approximately the same time as the corresponding dry properties in the first half of 1948. The dry and wet properties of Sisal, Henequen, domestic Abaca, and Sansevieria (Table IV) were determined in the last half of 1950—after about 2½ years of storage. Comparison of the dry data given for these latter fibers in Tables II and IV shows that following storage, the dry tenacity is unchanged and the elongation lowered, while the wet properties are substantially the same as the dry properties before storage, except for Henequen. For the Henequen fibers, the wet-out tenacity is lower and the elongation higher than the dry properties. These results suggest a hysteresis in moisture regain, since all dry tests were made following equilibrium at 70°F and 65% R.H. Active work is at present being prosecuted on the moisture-hysteresis aspects, since changes in properties resulting from storage appear to be significant in light of the stock-piling aspect of the cordage fibers. While no definite conclusions have yet been reached, it appears that following wetting-out of stored fibers and reconditioning to 70°F and 65% R.H. from the wet side, the original dry properties, as given in Table II, are found for all of the fibers, including Henequen. For example, following such a conditioning procedure, the tenacity of Victoria Henequen was found to be 3.3 g./den., and the elongation was 5.1%, both of which closely agree with the data for the unstored fiber of 3.2 g./den. and 5.1%, respectively.

It is planned to report on the aging aspects of the fibers from time to time in future papers.

Knot strength of hard fibers.—The knot strength of textile yarns and fibers has found great use as an index of the flexibility of such materials, one criterion being that a low knot strength indicates poor ability to resist the deformation imposed by the large transverse forces developed when a knotted member is stressed. Another criterion has been concerned with describing a low knot strength as being indicative of the brittleness of a material, in the sense that the physical process of tieing and tightening a knot produces rupture of the material in the knotted region.

The mechanism of failure of knotted members is an important part of this study, since cordage structures—in particular, light cordage—must be capable of withstanding knot stress without rupture. Hence, knot-strength evaluations have been made for Manila Abaca, Sisal, Henequen, and Sansevieria fibers.

TABLE V. KNOT TENACITY OF UNOILED HARD FIBERS

Fiber	Tenacity Knot* (g./den.)	Tenacity Straight (g./den.)	Knot efficiency† (%)	Elongation (straight break) (%)
Manila Abaca	1.48 (25)‡	6.71	22.1	2.65
Sisal	1.36 (22)	4.40	30.8	2.72
Henequen	1.36 (20)	3.30	41.1	4.77
Sansevieria	1.32 (27)	4.50	29.4	2.70

* Each value is the average of 125 breaks.
† Defined as ratio of knot tenacity to straight tenacity.
‡ Figures in parentheses are coefficients of variation (%).

With respect to technique of testing, long lengths of fiber were weighed, and an average denier determined. These fibers were cut in two, and the two halves were joined by knotting their mid-points by a binders knot. The free ends are capable of being stressed, and it is then possible to determine the tenacity of a knotted fiber.

The results of this study are given in Table V.

The data given in this table are important for the following reasons: (a) They illustrate the general poor knot efficiency of the hard fibers. At best, a knotted hard fiber cannot be expected to support more than about 40% of its unknotted strength, the knot efficiency for Henequen; (b) When tested in knotted form, the tenacities of the Sisals, Henequens, and Sansevieria are about 90% that of the Manila Abaca, indicating the superior knot efficiencies of these materials relative to straight tenacity over Manila Abaca; (c) The knot efficiencies given in Table V serve as a basis for explaining the knot efficiencies of cordage structures such as yarns made from these materials.

As stated previously, Sisals and Henequens find great use as baler and binder twine material. The principal loads to which such twines are subjected in use are knot stresses. It is clear from the results shown in Table V that similar resistance to knot failure would exist in equal-weight yarns of Henequen and Sisal. Hence, although for uses in which knotting is not required, a heavier Henequen yarn than Sisal would be required, the two yarns need only be of the same size in baler and binder twine. This would be true of Sansevieria fiber also. Manila, on the other hand, even though its knot efficiency is low, has a sufficiently high straight tenacity so that its knot tenacity exceeds that of the other fibers. How-

TABLE VI. EFFECTS OF TIEING KNOT ON TENSILE STRENGTH OF MANILA ABACA FIBER
(PRELOADED TO APPROXIMATELY 0.3 G./DEN.)

	Before knotting	After knotting and unknotting†
Tenacity (g./den.)	6.71	6.62*
Elongation to rupture	2.65	2.70*

* Each value is the average of 50 breaks.
† Knot efficiency is 22.4%.

ever, considering the low knot efficiency of Manila, it would undoubtedly be uneconomical to use this material in knotted form. The subject of the mechanism of knot failure has not yet been fully discussed, but the concept developed in this work is concerned with the tensile-stress concentrations developed in a structure, such as a knotted yarn or fiber, when these members are subjected to tensions, and their geometric paths involve a very low radius of curvature. These stress concentrations are not limited to knots but apply to any use wherein the path of the member is curved. Such uses occur, for example, in the case of cordage passing through sheaves, etc., and it would be expected, in general, that the relative efficiencies of the fibers in such uses could also be compared on the basis of the knot efficiencies given in Table V.

In attempting to explain the differences between the knot efficiencies given in Table V, it is interesting to compare the trend in knot efficiencies with the trend in elongations to rupture of the fibers, also shown in Table V. It is apparent that there is a perfect trend-correlation between these sets of values, although it is not linear. As will be shown later, a more logical basis for correlation exists in the amount of flow or creep in the fiber, as indicated by the shape of its stress-strain curve, but, for the purposes of the present discussion, it can be temporarily assumed that high extension to rupture is indicative of high knot efficiency of a hard fiber.

It now remains to be determined what part this high elongation to rupture plays in producing high knot efficiency. As stated previously, one criterion of knot strength is the possibility of rupture of parts of a fiber due to the process of tieing and tightening a knot, the "brittleness" of the fiber being responsible for the rupture. In order to study this possibility, samples of Manila Abaca fiber were knotted and loaded to 20% of their knot strength, and were then unknotted and subsequently tested in straight tension. The results of this study are given in Table VI.

It is clear from the data given in Table VI that the fibers are not damaged by the process of knotting and tightening, and, hence, the low knot efficiencies for Manila given in Table V cannot be explained by damage. Previous work done at the Fabric Research Laboratories showed these same conclusions to apply to Henequen fiber. Detailed discussion of a more probable mechanism of knot failure will be given in a subsequent paper.

Effects of twist on strength of hard fibers.—The work covered herein is a necessary part of the study of the translation of the properties of hard fibers into cordage structures. As is obvious, the tensile strength of a yarn depends, among other things, upon the strength of the individual fibers which compose the yarn *as they exist in the geometrical configuration of the yarn.*

From the geometry of twisted yarn structures, it is clear that each fiber is twisted about its own axis as it lies in the yarn. It is then important to know the effect of applying tensile loads to a twisted fiber, with particular emphasis on observing any changes in tensile strength resulting for such a condition. While one would not expect any change to occur in the case of the highly extensible synthetic fibers, the low elongation of the hard fibers could cause a loss in strength.

Fibers of Manila Abaca, Henequen, and Sisal were twisted varying amounts, and were tested while in a twisted condition, the axis of the helix corresponding to the axis of the straight line joining the fiber ends. Free contraction of the fiber was permitted during twisting. The results are given in Table VII.

Examination of the data given in Table VII shows that there is negligible difference between the tenacities of hard fibers whether they are in a twisted configuration or a straight one. Statistical analysis shows

TABLE VII. EFFECT OF TWIST ON TENACITY * OF UNOILED HARD FIBERS

Fiber twist (turns/in.)	Tenacity of: Manila Abaca (g./den.)	Sisal (g./den.)	Henequen (g./den.)
0	6.71 (22)†	4.40 (21)	3.30 (20)
0.6	6.50 (24)	4.20 (23)	3.24 (20)
1.2	6.60 (25)	4.40 (23)	3.20 (19)
1.8		4.40 (25)	3.24 (16)
2.4	6.70 (24)	4.32 (22)	3.15 (19)
3.0		4.35 (21)	3.15 (16)
3.6	5.80 (32)	4.20 (17)	3.18 (18)

* Each value, except those for zero twist, is the average of 50 breaks.
† Figures in parentheses are coefficients of variation (%).

that none of the tenacities given in Table VII for any twisted fiber differs significantly from that for any other twist for that fiber, including zero twist, except in the case of the 3.6 turns/in. Manila Abaca. Since the commercial twisting machines involved in hard-fiber processing produce twists not exceeding 2.5 turns/in., the data given in Table VII cover any feasible yarn twist. Hence, it can be concluded that within the limits of the commercially available twists, the strength of hard fibers is unchanged. This, of course, does not take into account any other processing effects which may occur simultaneously with twisting, such as abrasion or tearing of fibers.

Elastic properties of hard fibers, repeated stress.— Being composed chiefly of cellulose, the hard fibers exhibit the visco-elastic characteristics that are common to all high polymers. Methods for determining the effects of visco-elastic characteristics on the mechanical properties of filamentous materials have been described by Leaderman, Hamburger, etc. [4, 5, 6]. The procedure consists essentially of controlled alternate loading and unloading of a material up to various percentages of its breaking strength and noting the changes in the stress-strain diagrams.

This alternate loading and unloading should not be confused with a fatigue type of test. Its purpose is to determine chiefly such visco-elastic characteristics as the amount of permanent set (permanent change in length), the rate of recovery and elastic hysteresis, the change in energy absorption to rupture, etc.

Repeated-stress studies were carried out on unoiled Manila Abaca, Java Sisal, and Victoria Henequen. The maximum load, termed "conditioning load," was 90% of the breaking strength for the Manila Abaca, while tests on the Sisal and Henequen were carried out at two loads—50% and 90% of their respective breaking strengths. The results are given in Table VIII.

Considering the results at 50% of the ultimate load, statistical analysis shows that each property of Henequen differs significantly from the corresponding property of Sisal. It is apparent, then, from inspection of the data, that:

(a) Sisal fibers are more nearly perfectly elastic than are Henequen fibers. The higher value of the elastic performance coefficient [4] for Sisal is indicative of this fact, as is the difference in the amount of permanent set between the two fibers. However, it is to be noted that even though the amount of permanent set for Henequen exceeds that for the Sisal, the residual elongation remaining in the Henequen after repeated stressing exceeds that in the Sisal fibers.

(b) The elongation to rupture of Henequen is reduced from 4.77% (see Table III) to 4.38% as the result of repeated stressing at 50% of the ultimate load; while the elongations of the Sisal fibers are reduced from 2.72% to 2.28%. Both of these changes result from the removal of secondary creep. There were negligible changes in the tenacities of both of these fibers, and, hence, when energy absorptions are considered, Henequen still possesses a higher specific energy absorption (the energy absorption per unit gage length per denier) to rupture than does Sisal. These results indicate that if cordage structures were

TABLE VIII. ELASTIC PROPERTIES * OF UNOILED HARD FIBERS (REPEATED STRESS AT 50% AND 90% OF BREAKING STRENGTH)

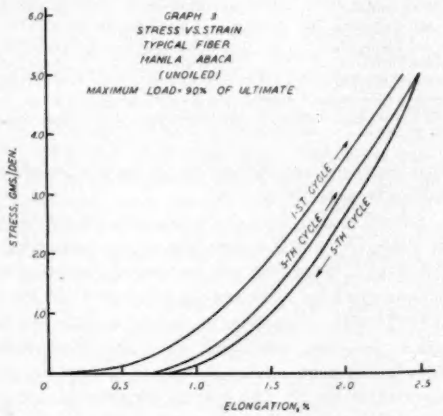
Fiber type	Conditioning load (% of ultimate load)	Permanent set (%)	Elastic performance coefficient	Residual elongation (%)	Tenacity (g./den.)	Energy absorption (g. cm./cm. den.)
Henequen Victoria	50	0.78 (33)†	0.52 (12)	4.38 (20)	3.44 (9)	95×10^{-3} (30)
Sisal Java	50	0.22 (57)	0.63 (27)	2.28 (12)	4.94 (11)	61×10^{-3} (21)
Henequen Victoria	90	1.91 (30)	0.39 (12)	3.31 (9)	3.75 (10)	74×10^{-3} (24)
Sisal Java	90	0.57 (24)	0.54 (11)	2.32 (12)	5.06 (10)	64×10^{-3} (22)
Abaca Manila	90	0.62 (21)	0.53 (15)	2.38 (8)	7.61 (9)	89×10^{-3} (17)

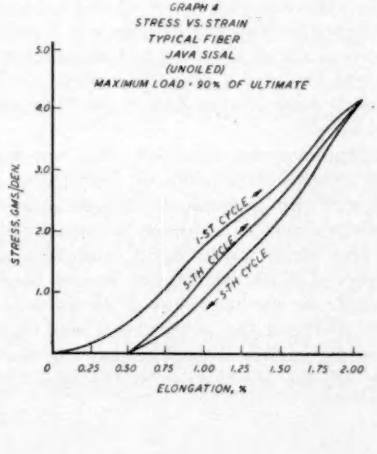
* Values for Henequen and Sisal are the averages of 10 tests; values for Abaca, the averages of 15 tests.
† Figures in parentheses are coefficients of variation (%).

designed with a factor of safety of 2, on a load-carrying basis, Henequen would be capable of withstanding approximately 55% more impact energy prior to failure than would Sisal.

Reference is now made to the 90% stress level, where the repeated-stress properties of Henequen, Manila Abaca, and Sisal fibers are compared for a mechanical conditioning load equal to 90% of the rupture load. The following observations can be made:

(a) It is clear that the amount of permanent deformation of Henequen exceeds that of Sisal and Manila. There is a negligible difference between the permanent sets of Manila and Sisal. The elastic performance coefficients for Manila and Sisal are equal and exceed that of Henequen.



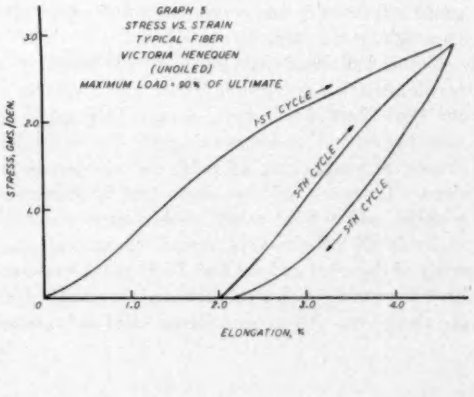


(b) The residual elongations of Manila and Sisal are both about the same, and show a negligible reduction from the elongation to rupture possessed by these fibers in a single loading to rupture. (See Table III.) Henequen, on the other hand, suffered a severe loss in elongation as a result of repeated stressing at 90% of ultimate load, the elongation decreasing from 4.77% to 3.31%.

(c) The change in the residual elongation reduced the energy absorption of Henequen so that following repeated stressing at 90% of ultimate load there was negligible difference between Sisal and Henequen fibers with respect to this property.

The importance of such data as those given in Table VIII cannot be overemphasized. From the point of view of the design of cordage structures, these data represent those constants of tensile performance which must be satisfied just so long as the cordage structure is to be used for more than one loading. It is true that compared to most synthetic fibers (*e.g.*, acetate or viscose) the absolute change in the mechanical properties of hard fibers is small. However, these changes must be considered in light of the properties of these hard fibers before repeated stressing, and, being relatively inextensible materials, the percent changes in the load-deformation characteristics are quite sizeable.

A word of caution must be inserted with respect to comparing the properties given in Table VIII with those in Table III. It is clear from the coefficients of variation given in Table III that the variability of the fiber properties is large. Obviously then, mechanical conditioning at 90% of the *average* breaking strength must necessarily involve testing those fibers the strengths of which are at least 90%



of the average. The properties of fibers of lower strength will not be present in any data which depend upon this given minimum strength. Hence, the statistical populations of those fibers the properties of which are summarized in Table III are not identical with those of the fibers the properties of which are given in Table VIII. Therefore, while it might seem that tenacities of the fibers increase as a result of repeated stressing, it can be shown that this could result from testing a stronger population of fibers in the repeated-stress studies.

This statistical condition is unavoidable. However, it does not preclude comparing the properties of the various hard fibers at equal mechanical conditioning loads, as was done in Table VIII. This is so since the coefficients of variation of mechanical properties of the three fibers studied in repeated stress are all just about the same (see Table III).

Typical repeated-stress curves for Manila Abaca, Java Sisal, and Victoria Henequen are given in Graphs 3, 4, and 5.

Effects of processing on Manila Abaca fiber.—

Oiling and combing: Prior to twisting the hard fibers into a yarn, an oiling and combing operation is employed. The purpose of the oiling is to lubricate the fiber surface for smoother manipulation of the stock during processing. The combing serves to parallelize and longitudinally randomize the fibers. A number of combings are consecutively made, the exact number being dependent upon the initial condition of the stock of fiber (*e.g.*, degree of entanglement of the fibers) and the final structure to be made from the fibers. The combs are actually a series of tapered pins attached to endless belts. The particular oil used for the pedigreed Manila Abaca fibers being studied was a mixture of wool grease and mineral oil, the gross add-on being controlled to approximately 12% by weight of the fiber.

To an individual accustomed to the handling of textile materials going into fabrics, the treatment of the hard fibers would seem severe. One would almost be certain that severe changes in the mechanical properties would occur following the combing operations. It was considered important to determine whether, and to what extent, such changes occurred. In Table IX are given the various mechanical properties of the oiled and combed fiber, and for convenience the corresponding properties of the unoiled fiber are also given. All stresses for the oiled and combed fiber are corrected for the 12% increase in weight resulting from the oil pickup.

TABLE IX. EFFECT OF OILING AND COMBING ON MECHANICAL PROPERTIES OF MANILA ABACA FIBER

	Unoiled fiber	Oiled and combed fiber
Tenacity (g./den.)	6.71 (21)*	6.90 (27)
Rupture elongation (%)	2.65 (22)	3.26 (19)
Energy absorption (g. cm./cm. den.)	78×10^{-3} (19)	100×10^{-3}
Knot tenacity (g./den.)	1.48 (25)	1.78 (20)
Knot efficiency (%)	22.4	25.6
Repeated-stress performance (90% of ultimate load):		
Permanent set (%)	0.62 (21)	0.54 (22)
Elastic performance coefficient	0.53 (15)	0.60 (20)
Residual elongation to rupture (%)	2.38 (8)	2.61 (10)
Tenacity (g./den.)	7.61 (9)	7.12 (14)
Energy absorption to rupture (g. cm./cm. den.)	89×10^{-3} (17)	100×10^{-3}
Tenacity when twisted:		
0 turns/in.	6.71 (22)	6.90 (27)
0.6 turns/in.	6.50 (24)	6.60 (20)
1.2 turns/in.	6.60 (25)	6.70 (27)
2.4 turns/in.	6.70 (24)	6.60 (31)
3.6 turns/in.	5.80 (32)	6.70 (30)

* Values in parentheses are coefficients of variation (%).

Statistical analyses performed on the data presented in Table IX indicate that most of the properties of the Manila Abaca fiber are unchanged because of oiling and combing. The only characteristics which are altered are the elongation to rupture and those properties that are functional with the elongation—namely, the energy absorption both before and after repeated stressing, the residual elongation, and the knot efficiency. All of these properties are greater for the oiled and combed fiber than for the unoiled. This is perhaps not surprising since it is possible to conceive of the oil as having partially penetrated the fiber and produced a plasticizing effect. It is surprising, however, that no damage due to the combing is evidenced.

Summarizing the effects of oiling and combing on the mechanical properties of Manila Abaca fiber, it appears that insignificant changes occur in all properties except the extension to rupture and those properties which are dependent upon the extension. Where statistically significant changes have been produced, the absolute values of the differences are small. Hence, it can be stated that with the exception of any surface changes produced by the lubricating oil, the fibers arrive at the stage of being

TABLE X. EFFECTS OF TWISTING OPERATION ON MECHANICAL PROPERTIES OF MANILA ABACA FIBER
(FIBERS TAKEN FROM STANDARD YARN: 11.0 TURNS/FT.; 300 FT./LB.)

	Original oiled and combed fiber	Fiber from inside of yarn	Fiber from outside of yarn
Tenacity (g./den.)	6.90 (27)*	6.70 (25)	5.90 (30)
Elongation to rupture (%)	3.26 (19)	3.13 (24)	2.96 (29)
Number of tests	100	350	350

* Values in parentheses are coefficients of variation (%).

twisted into yarn in substanially their original, unoiled condition.

Twisting: It has been shown that oiling and combing of Manila Abaca fibers do not produce any substantial changes in their mechanical properties, with the exception of a slight increase in the elongation to rupture. Prior to investigation of the translation of these fiber properties into yarn, it is important to inquire into the possible effects of the actual twisting operation on the mechanical properties. Although it was shown previously that when individual hard fibers are tested in a twisted condition they exhibited negligible changes in strength, the possibility of fiber damage which can occur simultaneously with the commercial twisting operation was not excluded.

In order to investigate twisting effects, fibers were selected from the standard Manila Abaca yarn going into ¾-in. rope. This yarn possesses approximately 11 turns/ft., weighs approximately 300 ft./lb., and was made from the same Manila Abaca fiber mix the properties of which have already been given.

It is apparent that one possible effect of processing can be any surface abrasion of the fibers which comprise the yarn. Even though there is considerable "darting" of fibers in and out of the yarns, it is possible to distinguish between those fibers which lie predominantly on the yarn surface and those which lie predominantly in a buried condition within the yarn. The mechanical properties of fibers selected from both of these yarn positions are shown in Table X.

It is clear from Table X that changes in fiber properties resulted from the twisting operation. Although there were negligible changes in the properties of the inside fibers, Table X shows that the outside fibers were damaged. With respect to tenacity, the outside fibers were 14½% weaker than the original oiled and combed fiber, while the elongation was reduced by 9%. These changes indicate a loss in energy absorption of 22% for the outside fibers.

It is interesting to inquire into the effects of the magnitude of the twist on the strength losses noted in Table X. While it was previously shown that the strength of the fibers that were tested while in a twisted condition did not vary with the twist, it is possible that, since only the surface fibers showed damage, any such processing effects, such as, say, surface abrasion, would affect different yarn twists to different magnitudes.

A series of yarns of different twists are being studied as part of the investigation concerned with rope geometry effects. While the standard yarn contained only Manila Abaca fiber, the yarns of different twists were taken from normal production of the Boston Navy Yard as of February, 1948. These yarns contained mixtures of 30% Philippine Abaca and 70% Costa Rican Abaca, and hence are not comparable with the standard yarn. *The properties of fibers from these yarns of different twists should therefore be regarded apart from the results given for the standard yarn.*

Statistical analysis of the data given in Table XI indicates that for both the inside and outside fibers the amount of yarn twist does not affect the fiber strengths. None of the inside-fiber tenacities given in the table differs significantly from any other in-

TABLE XI. MECHANICAL PROPERTIES* OF FIBERS FROM YARNS OF DIFFERENT TWISTS
(YARN WEIGHT 270 FT./LB.)

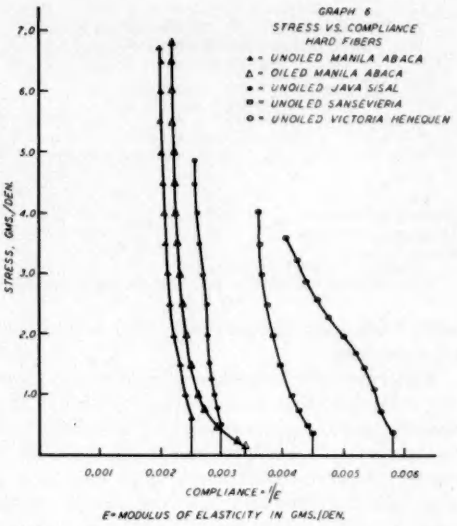
Yarn code number	Yarn twist (turns/ft.)	Fibers from inside of yarn: Tenacity (g./den.)	Fibers from inside of yarn: Elongation (%)	Fibers from outside of yarn: Tenacity (g./den.)	Fibers from outside of yarn: Elongation (%)
2A	10.6	6.00 (31)†	2.81 (21)	5.10 (31)	2.69 (24)
2B	11.1	5.65 (36)	2.89 (27)	6.46 (30)	3.21 (22)
2C	13.9	5.35 (43)	2.64 (30)	6.00 (37)	3.09 (32)
2D	17.2	6.51 (37)	3.26 (27)	5.15 (44)	2.66 (38)

* Each value is the average of 20 tests.
† Figures in parentheses are coefficients of variation (%).

side-fiber tenacity; of the outside fibers only those from yarns 2A and 2B are barely significantly different. With respect to elongations at rupture, the twist affected the inside fibers so that only fibers from yarns 2C and 2D are barely different; while for the outside fibers, only those from yarns 2A and 2B indicate possible significant difference from each other. Considering the great number of pairs of values in Table XI for comparison of differences, it is apparent that the magnitude of the yarn twist does not affect the strength and elongation of the fibers, either inside or outside.

With respect to the difference between the inside and outside fibers from yarns of varying twist, analysis of the data given in Table XI showed that neither the strengths nor elongations differ significantly. This result differs from that shown for the pedigreed control set of fiber and yarn in Table X, wherein it was indicated that for the standard yarn twist of 11.0 turns/ft. the outside fibers were significantly weaker than the inside fibers. Information received from the Boston Navy Yard revealed that quality-control figures for the production lot from which the yarns of varying twist were taken indicated poor uniformity and strength of cordage. Hence, it is extremely likely that any differences between inside and outside fibers for the data given in Table XI are masked by the extreme variability of the fibers, and a tremendous number of fiber tests would be required in order to state confidently whether there is or is not any difference in strength between inside and outside fibers. Rough calculations show that in order to indicate that fiber-strength differences of 14% are significant, approximately 100 tests of inside or outside fibers of the type shown in Table XI would be required. Considering the comments made by the Boston Navy Yard representatives, it is doubtful if the results of such a large-scale investigation would be of any worth. Instead, it is planned to obtain a new set of varying-twist yarns of normal quality variability for a re-evaluation of the effects of yarn twist on fiber properties. Such tests have recently been completed and they show that twisting of fibers into yarns does not damage the fibers.

Sonic compliance of hard fibers.—A technique has recently been developed for the determination by sonic methods of the moduli of elasticity of filamentous materials which exhibit visco-elastic characteristics [1, 5]. The modulus of elasticity, in addition to defining an important constant of the material which is necessary for engineering design, also defines an important fundamental physical property of the fiber which serves as a precise index of its degree of orientation and/or crystallinity. Materials with high moduli indicate high tensional stiffness, and conversely. For most filamentous materials which exhibit the visco-elastic characteristics of primary and secondary creep [5], it is found that more than one modulus exists—that is, the modulus of elasticity varies with the tensile load on the sample. Hence, the modulus must be specified by a plot of stress *vs.* modulus.



Rather than plotting directly stress *vs.* modulus, the inverse of the modulus, $1/E$, termed "compliance," is plotted *vs.* the stress. The higher the compliance, the greater the elastic extensibility of the material, and conversely.

Graph 6 shows the stress-compliance curves for a group of hard fibers, including unoiled Manila Abaca, Java Sisal, Victoria Henequen, and Sansevieria, and oiled Manila Abaca. Each value of compliance plotted represents an average of 10 determinations.

It is clear from Graph 6 that the various cordage fibers possess widely different compliances. Considering only the unoiled fibers, Victoria Henequen shows the greatest tensile extensibility, while Manila Abaca, which is the stiffest fiber, has a compliance which is only 43% that of the Henequen. Java Sisal has compliances which are relatively close to those

of Manila Abaca; the curve for Sansevieria falls between those for Java Sisal and Henequen. It is to be noted that the shapes of the stress-compliance curves are the same for all materials except the Victoria Henequen. The data given in Graph 6 seem to substantiate the differences in elastic properties amongst the hard fibers as described previously by the stress-strain data.

Graph 6 shows that oiling modifies the properties of Manila Abaca to a small degree. The higher compliance value for the oiled Manila, combined with the fact that except at very light loads the curves for the oiled and unoiled Manila are substantially parallel, indicates that the oil produces the slight plasticization of the fiber already described, but this is removable by stretching at very light loads.

Effects of low temperature on mechanical properties of Manila Abaca fiber.—Arctic operations by the Navy make mandatory a knowledge of the performance of cordage materials at low temperatures. During the initial stages of this investigation, a cold room at the Massachusetts Institute of Technology was made available to the Fabric Research Laboratories, Inc. Although time did not permit an exhaustive study, it was possible to make a limited investigation into the effects of very low temperature on both oiled Manila fibers and yarns. Since the cold room was in operation at $-70°F$, the data taken refer to that temperature.

The tenacity and elongation to rupture of oiled Manila Abaca fibers at $-70°F$ are compared with properties at $+70°F$ in Table XII.

It can be seen from these data that the properties of Manila Abaca fiber are severely impaired at $-70°F$. Statistical analysis showed that both the tenacities and elongations are highly significantly different. At $-70°F$ the tenacity of Manila Abaca fiber was reduced by 35%, and the elongation was reduced 43%. Although the energy absorption is not given at $-70°F$, the loss in energy absorption to rupture may be estimated to be about 63%, indicating that at $-70°F$ the impact-resistance of Manila Abaca fiber is greatly reduced.

TABLE XII. MECHANICAL PROPERTIES AT LOW TEMPERATURES OF OILED AND COMBED MANILA ABACA FIBER

	Tenacity (g./den.)	Rupture elongation (%)
Control (+70°F, 65% R.H.)	6.90 (27)*	3.26 (19)
Equilibrium at −70°F	4.46 (34)	1.86 (24)
Wet-out and frozen at −70°F	5.01 (46)	1.96 (35)
Wet-out, frozen (−70°F), and reconditioned to +70°F, 65% R.H.	6.65 (26)	2.71 (26)

* Figures in parentheses are coefficients of variation.

The moisture content of the fiber must be considered when low-temperature properties are being determined. The data in Table XII are for fibers with normal moisture regain at $-70°F$—that is, the fibers were allowed to come into equilibrium with the atmosphere of the cold room prior to testing. It is of interest to inquire into the effects of complete wetting-out of the fibers immediately before insertion into the cold room. The fibers then would contain frozen water, a condition which could easily duplicate those for Arctic uses of cordage. The results of testing wet-out fibers at $-70°F$ are given in Table XII.

Statistical analysis shows that there is no difference between the mechanical properties of the dry and wet fibers, indicating that if used dry or wet at low temperature, that part of the performance of cordage structures depending upon the mechanical properties of the fiber at that temperature will not be changed.

While it was not the purpose of this investigation to study the mechanism of changes in properties at low temperature, it is obvious that any information on causes for the loss in strength and elongation would be valuable. One mechanism involves the possible damage of fibers at low temperatures when whatever moisture they retain is frozen. In order to investigate this mechanism fibers were wet-out and frozen at $-70°F$, after which they were allowed to come into equilibrium at $+70°F$ and 65% R.H., and were subsequently tested. The resulting properties are given in Table XII.

It is shown that no damage of the fibers due to moisture freezing at $-70°F$ occurred. The tenacities shown in Table XII are insignificantly different, while the elongations of the reconditioned fibers, although different statistically, are nowhere near as low as those of the fibers tested at $-70°F$. In addition to disproving the mechanism of fiber damage, this result is important from the point of view that in use, after a single thawing, the mechanical properties of the fibers present in cordage structures will be substantially the same as their original properties. This is not meant to imply that the cordage structures will necessarily exhibit the same properties, since the influence of water becoming frozen *between* fibers

within, say, a yarn can conceivably produce damage of the fibers.

Summary

The mechanical properties of hard fibers which are deemed most significant in terms of cordage performance have been determined by means of analyses of tensional properties under various environmental and physical conditions, as the necessary first step toward the completion of the study of the effective utilization of all available supplies of cordage fiber.

Utilizing the new fiber-testing techniques, such as the Pulse Propagation method for measuring elastic moduli and constant-rate-of-elongation loading devices equipped with resistance wire strain gages, various grades and types of fiber, including Sansevieria, Henequen, Sisal, and Abaca, were studied. The influences of temperature, humidity, previous history of water treatment, fiber twist, knotting, fiber damage during manufacturing, fiber position in the yarn as it may influence damage, oiling and combing during manufacture, etc., on the tensile strength, elongation, and energy absorption were measured. Particular emphasis was placed on the study of Manila Abaca, since this fiber is most widely accepted as the best available raw material for rope.

In summation, it has been shown that:

1. The elongation properties of a cordage fiber must be considered to be factors of equal importance with strength properties in limiting the performance of a rope in shock-loading and in repeated-stress application, in so far as elongation and recovery modify the energy-absorbing characteristics of a fiber.

2. The full measure of the potential performance of a fiber is best defined by a measure of its energy absorption characteristics after the removal of secondary creep (nonrecoverable deformation).

3. The differences in elongation properties evidenced by the fibers studied, although being small in absolute numbers, are high percentage-wise and markedly influence energy absorption. This fact has largely been overlooked in the cordage industry, and the implications of these differences have only been made clear by the development of sensitive measuring devices and by the recognition of the full implications of energy-absorption concepts in understanding the behavior of textile structures.

Having tabulated this type of performance and having tabulated the modifying effects of moisture, temperature, twist, fiber damage, etc., the research is currently being directed toward the utilization of these basic data in the further study of the influence of yarn and rope geometry on the translation of these fiber properties as the next logical step in determining engineering design techniques for building a rope from a given raw material.

Acknowledgment

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Acknowledgment is also made to Mrs. Melissa Landing, Mrs. Barbara Dwyer, and Mrs. Patricia Carney for their painstaking efforts in the laboratory.

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An Electronic Work-Recovery Integrator

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THE ADVENT of electronic means of measuring stress and strain in the testing of fibers has brought forth several new instruments for a much greater variety of tests for the evaluation of the properties of fibers. One of these is the electronic work-recovery integrator. This instrument replaces the planimeter for measuring areas under the stress-strain curve. A much larger number of tests can be performed in considerably less time. The instrument is particularly adaptable to electronic testing systems using servo-driven recorders such as the Brown Electronik or the Leeds & Northrup Speedomax. A direct reading may be obtained on a simple microammeter as percent work recovery to within 1% at normal testing speeds. In this case the area under a curve during the loading cycle is measured and stored as a quantity of electricity. The area under the curve during unloading is likewise measured, and an indication is then easily obtained which is a direct ratio, in percent, of the work regained to the work expended on the sample.

This instrument is also useful for integrating any varying voltage with respect to time as long as the frequency of variation is not high. As the frequency increases, the accuracy of the measurement decreases. Provision is made for a wide variety of operating conditions. At practically all speeds encountered in physical testing, except impact testing, the electronic integrator can integrate a voltage, representing stress, with respect to time or to elongation to within 5% or less. A photograph of the electronic work-recovery integrator, showing the operating panel and part of the integrator chart, is shown in Figure 1.

Theoretical Considerations

The basic testing instrument for which this integrator was developed is comprised of a stress-measuring system using strain gages, a constant-rate-of-elongation drive for the sample, and an electronic recorder to measure and plot the curve of stress with respect to time. Since the sample is being elongated at a constant rate, strain may be substituted for time and the curve obtained will be a direct stress-strain curve. As the first step in measuring work recovery, it is then desired to integrate the area under this curve. Therefore, the instrument should perform the following mathematical function:

$$y = k \int_{t_1}^{t_2} \theta \, dt,$$

where y = output indication of the integrating instrument; k = a proportionality constant; θ = input to the instrument from the stress-measuring instrument; t_1 = time at start of integrating period; t_2 = time at end of integrating period.

Under these conditions the instrument would be simply a stress-time integrator and would be suitable for measuring the work, represented by the area under the curve from t_1 to t_2, during the loading period. If at regular intervals during the straining period an electrical charge which is proportional to the ordinate of this curve is injected into a capacitor, then the total charge collected by the capacitor at the end of the cycle will be proportional to the area under

FIG. 1. *Electronic work-recovery integrator, showing operating panel and recorder.*

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the curve. This charge may be directly measured by an infinite-impedance-input vacuum-tube voltmeter. By adjusting the sensitivity of the voltmeter system the charge on the capacitor can be set to read 100 on the indicating meter (standardizing). With the voltmeter then set at a particular sensitivity, the capacitor may be disconnected and the charge removed by shorting it. The instrument is then ready to measure percent work recovery.

A typical curve of the sort obtained in these measurements is shown in Figure 2. The quantity which the instrument is to measure may be expressed by the equation:

$$\% \text{ Work recovery} = \frac{\text{area } B}{\text{area } A} \times 100.$$

It should be noted that a pause (10 to 30 sec.) occurs at the end of the straining period. This allows sufficient time to perform the standardizing operation to which the measurement for the recovery period may be compared.

The integration of the area under the curve during the recovery period (area B) is accomplished in a similar manner. At the end of this period the voltmeter is again connected to the charge-collecting capacitor, and the reading obtained is the percent work recovery. This is true since the work expended in the straining period is represented as 100 on the meter.

The system for injecting charges that are proportional to stress utilizes a relaxation oscillator the frequency of which is *constant* at any preset value. This oscillator operates a relay about 100 times each during the measurement of area A and area B. The relay discharges a very small capacitor, charged to a voltage that is proportional to stress, into a very large capacitor. The values of these two capacitors are such that error due to voltage distribution between them will be less than 0.1% and constant at any preset value of oscillator frequency (since they constitute capacitive reactances). Ideally, it would be desirable to have the small capacitor transfer its *entire* charge to the large capacitor. However, this is impossible and some compromise must be reached between two factors—the ratio of capacitance values and the limiting factor of leakage-resistance.

Design Considerations

Essentially, then, this system for integrating area A and area B and providing a direct measurement of

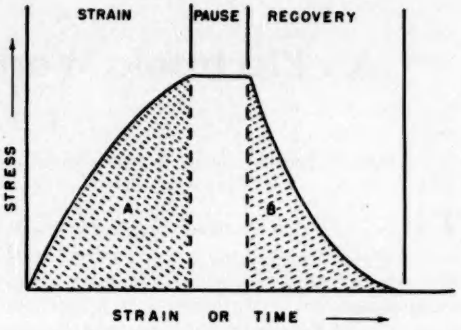

FIG. 2. *Typical stress-strain curve used to determine work recovery.*

work recovery consists of the following circuits: (1) a constant-frequency relaxation oscillator operating a suitable relay; (2) an input linear potentiometer to produce a voltage proportional to stress (curve ordinate); (3) two capacitors the ratio of capacitances of which is very large (preferably several thousand), the leakage-resistance of which is extremely high, and the charge of which can be completely dissipated by momentary shorting; (4) a vacuum-tube voltmeter with as near infinite input resistance as possible; (5) a calibrating and standardizing network; and (6) associated power supplies. These circuits are illustrated in Figure 3.

Because it would be practically impossible to introduce any form of heavy load, such as a switching relay, directly into the relaxation oscillator circuit, it is necessary to add a stage of amplification in order to operate the relay properly. A type 6SJ7 tube (industrial type 5693) was chosen for its high plate resistance. By experimental development it was found that a Clare relay No. 40FC (10,000 ohm-55,600 turns) operating a Microswitch with special polystyrene or low-leakage phenolic insulation (Cat. No. X3756) was necessary. It was also found that this oscillator and relay combination would not operate with the stability necessary much over 500 cycles or transfers per min. More than a single frequency was found to be necessary to cover the range of tests encountered. Therefore, a switching system should provide three frequencies (480, 240, and 120 cycles per min.).

A Helipot 10-turn potentiometer geared to the pen-drive of a servo-type rectilinear recorder provides a voltage that is proportional to the ordinate on

the chart (stress). Voltage is supplied by a separate regulated power supply in such a way as to allow an artificial zero setting anywhere up to 80% of the ordinate on the chart. This has been found to be necessary where sample tests begin at some point above the bottom of the chart. Three positive voltage values may be manually selected for this potentiometer (75, 150, and 300 v.).

The potentiometer voltage is fed to the normally closed terminal of the switching relay. The common terminal connects to the small capacitor, while the normally open contact connects to the large capacitor. The small capacitor is first charged to a voltage that is proportional to the ordinate of the stress-strain curve, and that small charge is then transferred to the large capacitor. Upon experimentation with this circuit it became apparent that "interfacial polarization" of the large capacitor would cause serious error in the measurement of the integral charge. "Interfacial polarization" is an undesirable phenomenon which is present when using ordinary paper or oil-impregnated dielectric capacitors. If a paper capacitor is charged to 300 v., for instance, and then momentarily shorted, the capacitor will exhibit a small "retained" charge upon opening the short circuit. This effect is not serious and is barely noticeable where polystyrene or polyethylene is used as the dielectric. From this standpoint, mica is also a suitable dielectric. Consequently, provision was made so that two small molded Bakelite capacitors may be selected which have mica dielectric. The very large capacitors use polyethylene as the dielectric and are further protected by moistureproof, hermetically-sealed cans.

The vacuum-tube voltmeter for this application has a range of 0 to 5 v. Within this range it must have good stability and as high input impedance as possible, since the charge on the large capacitor must be

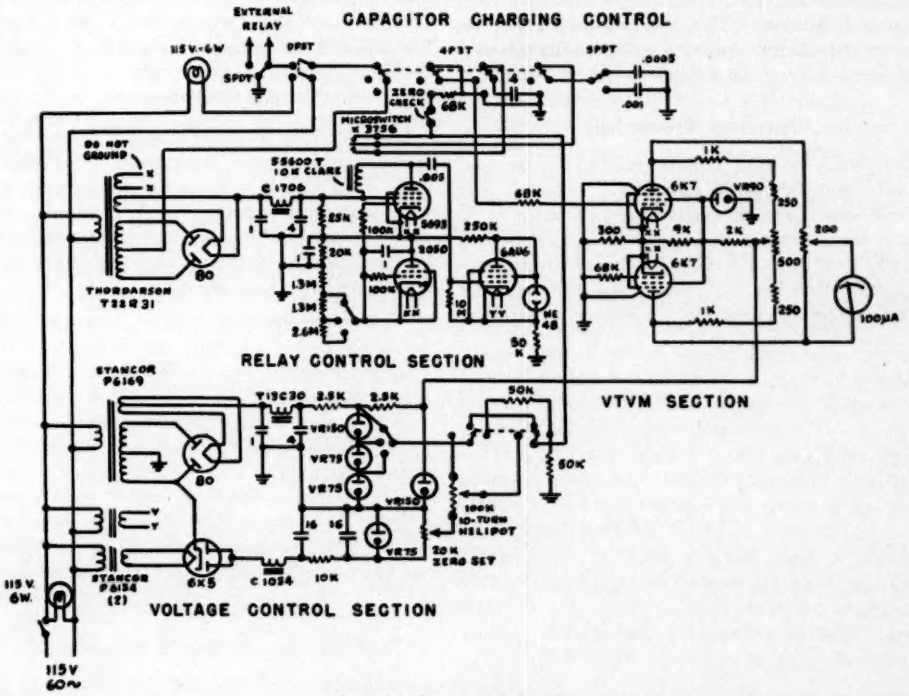

FIG. 3. *Integrator circuit.*

maintained accurately while its magnitude is being measured. A conventional balanced push-pull D.C. circuit was chosen for these characteristics.

It was considered desirable to have some form of network to determine quickly if the integrator is performing correctly. A switch is provided for selecting two voltages, one twice the other, to apply in place of a signal from the pickup potentiometer. By charging the storage capacitor with a fixed number of pulses from the higher voltage, X, and setting the vacuum-tube voltmeter on 100, and then shorting it and selecting the lower voltage, $X/2$, for a second charge using the same number of pulses, a 50% reading should be obtained.

A separate power supply is provided for the relaxation oscillator since any intercoupling with other parts of the unit would affect the oscillator frequency. Three rectifiers are then necessary to supply the oscillator, the voltage distribution system and the vacuum-tube voltmeter, and a low-voltage biasing system for zero setting on the recorder.

A separate circuit is provided for flashing a neon lamp with the relay. This is very helpful in operating the instrument. A 6AU6 tube prevents this system from affecting the stability of the oscillator.

Operation Precautions

To obtain the most accurate results, the vacuum-tube voltmeter should be calibrated. Since this type of voltmeter is not absolutely linear, the error at 50 (on a microammeter with linear calibration from 0 to 100) after the 0 and 100 points have been set, may be as much as 4 low. This varies too with the range being used on the vacuum-tube voltmeter. Therefore, the meter shunting rheostat should be calibrated in volts range. With very careful operation, results have often been obtained that are accurate to within .2%. The error obtained without these precautions will be between 2% and 4% at the center of the scale and 1% to 2% below 20 and above 80.

A minimum of 5 or 6 sec. is required to standardize the voltmeter at the end of the loading cycle. Usually, about 30-sec. delay is purposely used in tests where recovery from some strain below the breaking point is being studied. In cases of simple measurement of an area under a curve this delay is of no consequence and the initial reading may be taken in volts representing some value to which this figure will be proportional. For very long tests, a slow pulse rate and low charging voltage are used. Compromises must occasionally be made which affect the accuracy. Such would be the case of a rapidly fluctuating ordinate value over a long period where a high pulse rate may not be tolerated owing to the length of the test. The charge collected on the storage capacitor may otherwise be outside the range of the voltmeter. Of course, for special tests of this nature further development of the instrument would be required.

Acknowledgment

C. D. Wilson, formerly with the Pioneering Research Section, Rayon Department, Du Pont Company, did the basic work in the development of the work-recovery integrator. R. A. Beuerman, in the same section, has brought about several refinements by extending the operating range and improving the operability of the instrument.

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The Reaction of Combined Cystine of Wool with Sodium Bisulfite

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THE DISULFIDE cross-linkages in wool, due to the combined cystine, are of fundamental importance concerning the physical and mechanical properties of the wool. The changes in the elastic properties of wool, brought about by different reagents, are mainly due to the breaking of these disulfide cross-linkages.

If wool is treated with sodium bisulfite, according to Clarke [5] and Speakman [17], the combined cystine undergoes the following reaction:

$$R{-}S{-}S{-}R + NaHSO_3 \longrightarrow R{-}SH + R{-}S{-}SO_3Na.$$

In 1946, Carter, Middlebrook, and Phillips [4] summarized the results of previous investigations [7, 8, 12]. They came to the conclusion that not all of the combined cystine reacts in the same way with sodium bisulfite, but that cystine can be divided into different fractions. According to these authors, about 25% of the cystine does not react with sodium bisulfite, another 25% gives combined α-amino-acrylic acid, and one-half of the cystine is converted into cysteine and cysteine sulfonate side-chains. From the latter fraction part of the cystine can be restored by rinsing with water.

Moreover, Speakman [18] found that when human hair is reduced with sodium bisulfite the cystine cross-linkages can be restored by rinsing with a solution containing no oxidizing agent.

In connection with the extensive application of bisulfite for various treatments of wool, it is very important to know which reaction takes place in the wool and how this reaction can be influenced.

Therefore, we investigated the final effect of the reaction between wool and sodium bisulfite, and the influence of rinsing.

Experimental Procedure and Results

Samples of 400 mg. of woolen yarn, degreased by extraction with a mixture of trichloroethylene and ethanol, were first soaked for 30 min. at room temperature in 125 times their weight of 5% sodium bisulfite at pH 5.2,* and then reduced in an identical solution which had been preheated in a boiling water bath to 92°–95°C and kept at that temperature for 30 min. Heating the solution in a water bath prevented it from reaching the boiling point. Boiling had to be prevented because dispersion of tiny wool particles by the movement of the liquid might cause errors in the analytical data.

The bisulfite-treated wool was then subjected to the following aftertreatments: (*a*) no rinsing, immediate hydrolysis; (*b*) before hydrolysis, rinsing for 20 hrs. in 0.5% sodium acetate adjusted to pH 5.2 by the addition of acetic acid; (*c*) before hydrolysis, rinsing for 20 hrs. in 95% ethanol.

Two additional wool samples were reduced for 60 min. and two more for 90 min. In each case, one sample was hydrolyzed without rinsing and one was hydrolyzed after 20 hrs. of rinsing with the sodium acetate buffer solution.

The hydrolyses essential for determining cysteine and cystine were carried out in open test tubes with 10 ml. of 6*N* sulfuric acid. Hydrolyzing under CO_2 atmosphere did not make any difference. Although after 5 hrs. of boiling the keratin had not been completely hydrolyzed to amino acids, hydrolyzing for more than 5 hrs. caused no change in the values found for cysteine and cystine.

The amounts of cysteine and cystine were determined according to a method indicated by Shinohara [16], which enables cysteine and cystine to be determined simultaneously.

We started with three equal parts of the hydrolyzate, and adjusted them to pH 5.2 with sodium acetate and acetic acid. To the first part an aqueous solution of $HgCl_2$ and phosphotungstic acid reagent (Folin and Marenzi [10]) was added. The color of

* This solution was maintained throughout the investigation because of its optimum reducing action.

TABLE I

	Cysteine S (%)	Cystine S (%)	Cysteine S + cystine S (%)
Untreated wool	—	3.04	3.04
Wool reduced in 5% $NaHSO_3$ for 30 min.			
(*a*) Unrinsed	1.43	1.68	3.11
(*b*) Rinsed in buffer, pH 5.2, for 20 hrs.	0.03	3.04	3.07
(*c*) Rinsed in 95% ethanol for 20 hrs.	1.64	1.35	2.99
Wool reduced in 5% $NaHSO_3$ for 60 min.			
(*a*) Unrinsed	1.48	1.64	3.12
(*b*) Rinsed in buffer, pH 5.2, for 20 hrs.	0.14	2.88	3.02
Wool reduced in 5% $NaHSO_3$ for 90 min.			
(*a*) Unrinsed	1.44	1.67	3.11
(*b*) Rinsed in buffer, pH 5.2, for 20 hrs.	0.21	2.61	2.82

this solution served as a blank. To the second part only phosphotungstic acid was added. With the aid of a standard calibration curve the cysteine content was calculated from the measured color intensity. To the third part sodium bisulfite and phosphotungstic acid were added; then, with the aid of the blank, the known cysteine content, and the standard calibration curve, the cystine content was calculated from the color intensity caused by this reaction.

The cysteine and cystine percentages were determined for the wool samples treated with bisulfite in the above manner. The results are given in Table I.

Table I shows that after reduction for 30 min. about one-half of the cystine was converted. If, however, after the reduction the sample is rinsed with pH 5.2 buffer, it appears that cysteine is almost completely reconverted into cystine. Rinsing with ethanol does not cause reconversion of cysteine into cystine; on the contrary, the reduction of cystine appears to continue.

After more intensive reduction—namely, for 60 or 90 min.—rinsing again caused a considerable reconversion into cystine. However, a small amount of cysteine appears to be irreversible. This amount increases with a longer reduction time, being greater after 90 min. treatment than after 60 min.

TABLE II

	Cysteine S (%)	Cystine S (%)	Cysteine S + cystine S (%)
Untreated wool	—	3.07	3.07
Wool reduced in 5% $NaHSO_3$ for 45 min.			
(*a*) Unrinsed	1.91	1.15	3.06
(*b*) Rinsed in buffer, pH 5.2, for 20 hrs.	0.39	2.58	2.97
(*c*) Rinsed in 95% ethanol for 20 hrs.	1.35	1.74	3.09
(*d*) Unrinsed, but treated with monoiodoacetic acid, pH 8.3, for 15 min. before hydrolyzing	1.04*	1.10	3.18

* This value must be multiplied by 2 in order to obtain the actual cysteine S content (see text).

Additional samples of wool yarn were reduced for 45 min. Samples that were not rinsed after the reduction were immediately hydrolyzed. It appears from Table II that about two-thirds of the cystine was reduced, the cysteine S content being 1.91%. By rinsing after reduction in the pH 5.2 buffer the cystine percentage increased, but 0.39% cysteine S could not be reconverted into cystine. Also, it appears that rinsing with ethanol hampers the reconversion into cystine.

A few reduced samples were treated with monoiodoacetic acid of pH 8.3 for 15 min. at 95°C. According to Sanford and Humoller [15] and Mirsky and Anson [13] the free thiol groups are blocked by this treatment.

The color intensity measured is due to cysteine formed during hydrolysis from the cysteine sulfonate groups. As there is an equal amount of cysteine blocked by acetic acid groups, the value found has to be increased twofold. The result of the measurement was 1.04%; according to this method, therefore, 2.08% cysteine S must have been present.

It is also possible to block the thiol groups with the aid of ethyl iodide [13, 15]. To enable a comparison with the previous treatment, the reduced wool was shaken for 18 hrs. in a suspension of ethyl iodide in water at room temperature. The values found for the cysteine S and cystine S contents showed a large divergence. It appeared, however, that much more cysteine was reconverted into cystine than with the monoiodoacetic acid treatment.

TABLE III

	Cysteine S (%)	Cystine S (%)	Cysteine S + cystine S (%)
Wool treated with 0.1*M* KCN	nil	1.50	1.50
After reducing for 45 min. in 5% $NaHSO_3$			
(*a*) Unrinsed	0.52	0.98	1.50
(*b*) Rinsed in pH 5.2 buffer for 72 hrs.	trace	1.51	1.51

Cuthbertson and Phillips [6] observed that in wool treated with a potassium cyanide solution the combined cystine is converted into combined lanthionine:

$$R{-}S{-}S{-}R + KCN \longrightarrow R{-}S{-}K + R{-}S{-}CN \longrightarrow R{-}S{-}R + KCNS.$$

In order to examine the effect of this reaction we treated wool with potassium cyanide, as described by Farnworth, Neish, and Speakman [9].

Wool with a cystine content of 3.07% was treated with 30 times its weight of a 0.1*M* potassium cyanide solution at 66°C for 2½ hrs. After this treatment the wool still contained 2.53% sulfur (determined by Blackburn's method [2]); a determination of the cystine content showed that 1.50% cystine S was still present. This wool was then reduced for 45 min. in 5% sodium bisulfite. The results are given in Table III.

From Table III it appears that one-third of the disulfide S was reduced to cysteine. After prolonged rinsing in the buffer solution practically all the cysteine had been reconverted into cystine.

Woolen yarn was treated in the usual method with potassium cyanide for various reaction times. As the duration of the treatment was prolonged, the amount of nonreduced cystine gradually approached a certain final value. After a period of 16½ hrs. the wool still contained 0.91% cystine S. A few samples of this wool were then, as in the previous experiment, reduced in 5% sodium bisulfite for 45 min. The results are given in Table IV.

Table IV shows that in neither case (*a*) nor (*b*) was cysteine S present, and no change in the cystine S content occurred.

TABLE IV

	Cysteine S (%)	Cystine S (%)
Wool treated with 0.1*M* KCN for 16½ hrs.	nil	0.91
After reducing for 45 min. in 5% $NaHSO_3$		
(*a*) Unrinsed	nil	0.95
(*b*) Rinsed in pH 5.2 buffer for 18 hrs.	nil	0.91

Discussion

If wool is treated with sodium bisulfite, the disulfide cross-linkages are affected. In the opinion of Clarke [5] and Speakman [17] the combined cystine is decomposed into cysteine and cysteine sulfonate:

$$R{-}S{-}S{-}R + NaHSO_3 \longrightarrow R{-}SH + R{-}S{-}SO_3Na. \quad (1)$$

Hydrolysis of this bisulfite-treated wool causes a decomposition of the cysteine sulfonate into cysteine and sodium bisulfate:

$$R{-}S{-}SO_3Na + H_2O \xrightarrow{H_2SO_4} R{-}SH + NaHSO_4. \quad (2)$$

Reactions (1) and (2) occur when bisulfite-treated wool is hydrolyzed without previous rinsing (see Table I (*a*)). About one-half of the cystine is converted into cysteine.

However, if before hydrolyzing the reduced wool is rinsed in a buffer solution, cysteine is reconverted into cystine (see Table I (*b*)). This phenomenon can be explained by assuming that reaction (1) is an equilibrium:

$$R{-}S{-}S{-}R + NaHSO_3 \rightleftharpoons R{-}SH + R{-}S{-}SO_3Na.$$

By rinsing in water, bisulfite is withdrawn from the reaction, thus shifting the equilibrium to the side of cystine.

Rinsing in ethanol does not shift the equilibrium in favor of cystine (see Table I (*c*)), the solubility of sodium bisulfite in ethanol being very low.

The assumption of the existence of an equilibrum is in close agreement with the result of Katz and Tobolsky [11]. These authors studied the relaxation of wool fibers in water, bisulfite, etc. For fibers treated with bisulfite, the rate of relaxation appears to be much greater than that for fibers immersed in water. If the bisulfite-treated fibers are rinsed before stretching, the rate of relaxation is equal to that of the untreated fibers. In the case of the increased rate of relaxation, a great part of the disulfide cross-link-

ages are broken. These linkages are recovered by rinsing, and the rate of relaxation then decreases to that of untreated wool.

Table II shows that a higher cysteine percentage was found after treatment with monoiodoacetic acid. This phenomenon can be explained by assuming that at the beginning of the reaction with monoiodoacetic acid the concentration of bisulfite in the fiber is still high. The rinsing effect during this short reaction time (15 min.), however, is small. This results in a continuing reduction, causing a somewhat higher cysteine content.

By assuming an equilibrium, it can also be seen why so much cysteine is reconverted into cystine after the ethyl iodide treatment, for in this case the wool has been exposed to a certain rinsing effect for 18 hrs.

After an intensive reduction and subsequent rinsing in pH 5.2 buffer a small part of the cysteine and cysteine sulfonate is not reconverted into cystine (see Table II (*b*)), but remains in the form of cysteine. This phenomenon led to the conclusion that the wool disintegrated to such an extent that some cysteine groups were not able to react with cysteine sulfonate groups in order to form combined cystine and sodium bisulfite. This may be explained by assuming displacement of the corresponding cysteine and cysteine sulfonate groups so that they are beyond the reach of each other.

This conception is supported by the results of the reduction of KCN-treated wool. Here, all disulfide cross-linkages are re-formed because the more stable lanthionine groups prevent the wool from disintegrating.

Brown and Harris [3] observed a similar phenomenon when wool was reduced with sodium hydrosulfite and new cross-linkages were subsequently formed on treatment with alkyl halides. If there was some lapse of time between these treatments, the wool appeared to be badly damaged, as the newly formed cross-linkages were insufficient in number. However, if the treatments were carried out almost simultaneously, the combined cysteine groups had no opportunity to shift, and damage was avoided.

From Table IV it is apparent that the cystine fraction remaining after prolonged treatment with potassium cyanide, being about 30% of the original cystine content, cannot be reduced by sodium bisulfite.

This means that the cystine groups which could not be changed by potassium cyanide were also unaffected by sodium bisulfite. This cannot be a matter of equilibrium but must be due to the structure of keratin.

Thus, we conclude that the reaction of the combined cystine with sodium bisulfite must be considered to be an equilibrium reaction; also, it is probable that not all cystine groups are equally reactive. This assumption holds for the reaction with sodium bisulfite as well as for that with potassium cyanide and probably also for the reaction with thioglycolic acid [14]. This difference in reactivity must be attributed to a difference in accessibility of the keratin. For various reagents the accessibility has not the same magnitude. Alexander, Hudson, and Fox [1] studied the reaction of oxidizing reagents with combined cystine of wool and observed that different amounts of cystine were oxidized by $KMnO_4$, peracetic acid, and chlorine. There is a great similarity between these results and the estimations of the amount of crystalline matter in cellulose. Using chemical methods, the degree of crystallinity observed depends upon the method. Hence, in the case of cellulose it is also better to speak about accessibility.

The accessibility of wool for sodium bisulfite and for potassium cyanide is about the same. With both reagents, about two-thirds of the combined cystine can be converted, whereas one-third is not affected.

It seems possible that, under certain conditions, the combined cystine forms combined α-aminoacrylic acid by splitting off hydrogen sulfide. However, we did not find any indication of this reaction taking place. In a few cases the sum of the cysteine S and cystine S contents was, after the treatments of reducing and hydrolyzing, somewhat lower than the original cystine S content of wool, but the difference never exceeded 10%. This difference need not be caused by the formation of α-aminoacrylic acid, but may be due to other causes—for instance, the formation of lanthionine.

Conclusions

1. The effect of sodium bisulfite on wool can be represented by the reaction

$$R—S—S—R + NaHSO_3 \rightleftharpoons R—SH + R—S—SO_3Na$$

2. At 95°C about two-thirds of the disulfide cross-linkages of the wool react according to this equation. By rinsing in water, all of these cross-linkages are re-formed. The remaining one-third does not react with sodium bisulfite at all.

3. After a prolonged reduction the rinsing does not reconvert all of the cysteine and cysteine sulfonate into combined cystine. This phenomenon is due to molecular shiftings in the fiber, which prevent the reformation of some of the disulfide cross-linkages.

4. The cystine groups which cannot react with potassium cyanide to form lanthionine do not react with sodium bisulfite either. This may be explained by assuming different accessibilities for different parts of the keratin fiber.

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Letters to the Editor

Short communications in the form of Letters to the Editor are intended to provide prompt publication of significant new research results and to permit an exchange of views on papers previously published in the JOURNAL. These communications are not submitted to formal review as are research papers, and the editors do not assume any share of the author's responsibility for the information given or the opinions expressed. When work previously published in the JOURNAL is the subject of critical comment, the authors of the original paper are given an opportunity to submit a reply, which will be published concurrently when possible.

Rapid Dispersion of Cellulose in Cupriethylenediamine

AMERICAN ENKA CORPORATION,
RESEARCH DEPARTMENT
Enka, North Carolina
February 13, 1951

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

Solutions of cupriethylenediamine (*CED*) are widely used to disperse cellulose samples for viscosity measurements and D.P. determinations. If this solvent, in the usual concentration (0.5*M* in copper), is added directly to the sample, glutinous lumps that are very hard to disperse are frequently formed, especially if the D.P. is high. In order to avoid this difficulty and the prolonged stirring resulting from it, two solutions of *CED* are sometimes used, one (0.167*M* in copper) being added first to wet and partly swell the sample, the other (1.000*M*) being added later to effect complete solution. The volume of each solution is chosen to give a final copper concentration of 0.5*M*.

The following procedure is recommended for its simplicity and speed, solution of even high-D.P. samples being complete in 3–4 min. or less. The sample is prepared by cutting or tearing it into small bits, or (preferably) by passing it through a Wiley mill with a coarse screen. An accurately weighed portion is placed in a 25-ml. test tube, and from a pipet is added 10.0 ml. distilled water containing a small amount of wetting agent. (Several agents have been tried, such as Aerosol OT, Duponol ME, and Triton NE; all seem equally good, without effect on the viscosity. The concentration is not critical, 0.04% being used.) The test tube is shaken to ensure thorough wetting of the sample, and then 10.0 ml. of the stock *CED* solution (1.00*M* in copper) is added from pipet or buret. The tube is immediately closed with a rubber stopper, shaken vigorously for 15–20 sec., allowed to stand 30 sec. or longer (as convenient), and again shaken vigorously. For high-D.P. materials, further shaking may be required, but more than four shakings have not been found necessary. The solution is then ready for transfer to a viscometer. (If too large a sample, for its D.P., has been taken, the viscosity will be too great to permit rapid or complete solution; in such a case, it is best to start again with a smaller sample.)

Degradation of cellulose in *CED* is slow, and the time needed for solution by this procedure is so short that appreciable degradation does not occur before the viscosity measurement is complete, unless, perhaps, unusually high accuracy is required. Furthermore, though the viscosity is usually determined soon after making up the solution, delay is not serious if the test tube remains tightly stoppered, as the following tests show. Three equal weights of a cellulose sample were dissolved at one time, according to the above procedure. One solution was tested after standing 1 day, the second after 2 days, the third after 1 week. At the time of each test, as a control a freshly prepared solution was also tested, thus checking not only the stability of the solutions but

also the reproducibility of the method. The following results were obtained:

	Viscosity of aged solutions	Viscosity of controls
After 1 day	5.13	5.23
After 2 days	5.12	5.21
After 1 week	5.08	5.22

Even after a week, the viscosity is lower than that of a freshly prepared control by less than 3%.

When many samples must be tested, this simplified procedure, requiring only test tubes and a pipet, is especially helpful.

CHARLES H. LINDSLEY

The Effect of Gage Length on Fiber Strength Measurements*

THE GOODYEAR TIRE & RUBBER CO.,
RESEARCH LABORATORY
Akron 16, Ohio
March 26, 1951

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

Due to the current interest in the effect of gage length on the strength measurements of cotton fibers, I would like to review briefly some data obtained in this laboratory, relative to this problem.

In a previous article [2] which appeared in the JOURNAL a test procedure was described which permitted the testing of cotton fiber bundles at different gage lengths and at a test speed comparable to that used for most cotton end-products. In this test procedure the Pressley fiber jaws were mounted on the Scott IP–4 machine. Data were presented which compared the strength of cotton fiber with that of staple rayon at zero, 2.5-mm., 5.0-mm., and 7.5-mm. gage lengths. Comparisons were also made between the strength of cotton fiber and other natural, animal, and man-made fibers when tested at zero and 5-mm. gage lengths. It was pointed out that the strength values of fibers tested at 5-mm. gage length are approximately 50% below the strength values obtained at zero gage length. The apparent loss in strength of cotton fiber when tested at 5-mm. gage length was far greater than that of any of the other fibers reported. Because of this it was postulated that cotton fibers contain many more flaws and weak spots than any of the other fibers reported, in accordance with the "weakest link" theory [1].

Additional tests were made and reported in the JOURNAL [3] which showed that the Pressley tester, because of its higher test speed, registered strength values on cotton fiber approximately 12% higher than did the slower-operating Scott IP–4 machine. However, when both machines were operated at the same test speed, comparable strength values were obtained. It is apparent that because of the difference in test speeds between the Pressley tester and the Scott IP–4 machine and because of the drastic loss in the apparent strength of cotton fiber when tested at 5-mm. gage length, the 5-mm.-gage-length test will produce strength values on cotton fiber which are less than 50% of those obtained on the conventional Pressley tester.

The problem is to determine which test procedure will give tensile-strength values that are more comparable to those realized in actual service of the cotton end-product. For a partial answer to this problem let us examine some typical cotton tire cords.

* Contribution No. 176 from The Goodyear Tire & Rubber Co., Research Laboratory.

TABLE I. RELATION OF FIBER STRENGTH TO CORD STRENGTH

Cord No.	Fiber tenacity (Zero gage length) (g./den.)	(5-mm. gage length) (g./den.)	Cord tenacity (10-in. length) (g./den.)
1	4.74	2.64	2.37
2	4.27	2.24	2.26
3	4.55	2.21	2.14
4	3.62	2.13	1.96
5	3.93	2.06	1.84
6	4.02	1.94	1.95

TABLE II. RELATION OF FIBER STRENGTH TO WOVEN FABRIC AND YARN STRENGTH

Type of test	Strip width	Gage length	Tenacity (g./den.)
Fabric	2 in.	3 in.	2.42
Fabric	1 in.	3 in.	2.48
Fabric	1 in.	5 mm.	2.84
Yarn	—	10 in.	2.14
Yarn	—	3 in.	2.24
Yarn	—	5 mm.	2.86
Fiber	Bundle	5 mm.	2.99
Fiber	Bundle	Zero	4.56

Table I shows the strength of 6 cotton tire cords and the strength of the cotton fibers combed from the individual cords at both zero and 5-mm. gage lengths when tested on the Scott IP–4 machine [4]. Obviously, the 5-mm.-gage-length fiber test values correspond more closely with the cord strength (tested in the conventional manner) than do the zero-gage-length values. Had the zero-gage-length fiber tests been made on the higher-speed Pressley tester, the difference between the fiber strength and the cord strength would have been still greater. From the zero-gage-length test on the fibers combed from the cords it is evident that the high fiber strength is still present, yet in no manner can this high fiber strength be translated to the cord and be realized in service because there are apparently very few conditions under which stresses are applied to the cord at zero gage length.

Let us examine the data on woven cotton fabric shown in Table II [4].

The woven fabric, yarns raveled from the fabric, and fibers combed from the individual yarns were all tested at several gage lengths. Here, again, the 5-mm.-gage-length fiber tensile values correspond more closely with those for both the woven fabric and the individual yarns than do the zero-gage-length fiber tensile values. The zero-gage-length fiber tensile values are high, and, as in the case of cords, show that the conventional Pressley test procedure produces fictitious strength values for cotton fiber which cannot be translated into an end-product in such a manner that this high fiber strength can be utilized.

Further substantiation of the applicability of the 5-mm.-gage-length test procedure was obtained through the services of an experienced cotton classer [4]. A number of cotton samples which had previously been tested at both zero and 5-mm. gage lengths were given to the classer for his strength ratings. In all cases, with the exception of a few borderline samples, the classer's strength ratings agreed with the 5-mm.-gage-length tensile values but not necessarily with the zero-gage-length values.

Additional tests have shown that the percent loss in strength of cotton when tested at 5-mm. gage length as compared with zero gage length is not the same for all cotton varieties [4]. In some cases there have been complete reversals of strength ratings of different varieties by the 5-mm.-gage-length test over the conventional zero-gage-length test. Assuming that the 5-mm.-gage-length fiber test is a more reliable measure of the ultimate strength to be attained in a finished product, it seems logical that the cotton breeder who relies upon the Pressley tester may discard some very promising varieties in his screening tests.

It is not the intention of this laboratory to condemn the Pressley tester, but, rather, to urge certain modifications of the machine and of our present method of testing cotton fiber strength in general to permit the testing of cotton fiber under conditions which approximate service conditions of the finished products. Without question, a test procedure for cotton fiber strength showing approximately what ultimate strength can be realized in a subsequent end-product would be of great value to the cotton industry as a whole. We feel that the 5-mm.-gage-length test procedure accomplishes this objective.

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J. K. PHILLIPS

Copper Numbers of Methanolyzed Celluloses

FRICK CHEMICAL LABORATORY,
PRINCETON UNIVERSITY, AND
TEXTILE RESEARCH INSTITUTE
Princeton, New Jersey
March 10, 1951

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

In a recent communication, "The Reaction Between Methanol and Hydrogen Chloride at 0°C," Reeves and coworkers [1] state that in the work of Mehta and Pacsu [2] "high copper numbers were observed for the methanolyzed celluloses." This statement, as well as the subsequent suggestion that "this reducing action may have resulted from a side reaction—namely, hydrolysis by the water formed in the reagent during the prolonged treatment of the cellulose," is evidently based on their misinterpretation of our data, which are given as "copper numbers (ml. of 0.0416*N* $KMnO_4$ per g. material)," and not as conventional copper numbers. By conversion of the former into the latter the following figures are obtained for the more important samples:

Sample	Copper number
Native cotton (D.P. ~ 3,000)	0.04
Hydrocellulose (D.P. ~ 260)	2.75
Methanolyzed native cotton (D.P. ~ 260)	0.25
Methanolyzed native cotton (D.P. ~ 260) after hydrolysis with 0.05*N* HCl at 0°C for 48 hrs.	1.28

Since by direct estimations the authors found, respectively, only 0.0, 0.13, and 0.25 mol of water gained in the 7 days' old 4.1, 10.4, and 12.0 molar methanolic hydrogen chloride solutions, the writer is of the opinion that such small amounts of water will not seriously interfere with the rate of methanolysis which, in the case of simple glucosides cited by Reeves and coworkers [3] in connection with their similar findings on cellulose, "proceeds as much as one hundred times more rapidly than hydrolysis."

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EUGENE PACSU

The Nature of the Methoxyl Groups in Methanolyzed Celluloses

FRICK CHEMICAL LABORATORY,
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March 8, 1951

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

In the article "Glycosidic Methoxyl Groups in Methanolyzed Cellulose," TEXTILE RESEARCH JOURNAL, Feb. 1951, Reeves, Mazzeno, and Hoffpauir arrived at the conclusion that the acid-labile methoxyl groups in methanolyzed cellulose are linked in glycosidic combination (1 per molecule) and not in the acetal structure (2 per molecule) previously proposed by Mehta and Pacsu [5]. Reeves and coworkers' conclusion was based on the interpretation of their experimental results along three lines of investigation: retention of methoxyl after glycol cleavage; fractionation to approximately equal number- and weight-average molecular sizes; and hydrolysis of methoxyl groups from methanolyzed celluloses.

General Comments

At present it is a well-established experimental fact that the mild, heterogeneous, acidic degradation of the various celluloses proceeds with an initially rapid rate, the value of which constantly diminishes until it appears to reach zero at the stage of production of "limit hydrocelluloses," first so named by the present writer and his collaborators [4, 5, 6, 9]. This behavior is incompatible with the concept of a simple, linear macromolecule with "uniform and equivalent, covalent glycosidic linkages" without the aid of some *ad hoc* hypotheses. As a logical development of various modifying theories advanced by a host of investigators in the last 15 years, the writer has proposed [10] a structural formula for cellulose in which acetal linkages in equidistant, open-chain units were substituted for Haworth's ill-defined "lateral polymeric links." It was illustrated on this new formula that in the first stage of acidic degradation "probably the 1,4-acetal bonds across the laminae are cleaved resulting in the scission of the chains and production of shorter laminae ending in units with hemiacetal links." Cleavage of both the 1,5- and 1,4-acetal bonds of the open-chain units would result in the formation of "aldehydrol" end-groups which, by loss of a water molecule, could easily revert to the normal, pyranoid-ring structure. Application of this mechanism of hydrolytic attack to methanolytic degradation theoretically should give rise to chains with end-groups —CH(OH)OCH_3, —CH(OCH_3)$_2$, and possibly —CH(OCH_3)—O— and —CH(OH)—O—, as the primary products of the reaction. The problem of the nature of the methoxyl groups in the methanolyzed products, however, becomes more difficult if one recognizes the generic interrelation of methyl furanosides, pyranosides, open-chain hemiacetals, and dimethyl acetals in methanolic hydrogen chloride. Transformation of open-chain sugar dimethyl acetals into the anomeric furanosides and pyranosides [7, 13], and reversion of methyl glycofuranosides into pyranosides [2] have already been demonstrated. While the formation of furanoid end-groups in the methanolysis of cellulose must be discounted because of lack of chemical evidence for the presence of glycosidic bonds other than 1,4, the secondary formation of methyl glycopyranoside end-groups from primary degradation products with methyl hemiacetal, dimethyl acetal, or even free reducing end-groups remains a clear possibility. For this reason, the possible presence of some methyl glycopyranoside end-groups in methanolyzed cellulose samples cannot be construed as absolute evidence in favor of the linear concept of cellulose structure, nor can it be used as an argument against the theory of acid-sensitive lateral links in the cellulose molecule. On the other hand, it appears most unlikely that methoxyl groups of open-chain hemiacetal or dimethyl acetal character could secondarily form from primary scission products with methyl glycopyranoside end-groups as a result of an equilibrium reaction. Hence, the presence of methoxyl groups of the former nature in the products of methanolytic degradation may be regarded as *prima-facie* evidence in favor of the proposed structure of cellulose.

Comments in Detail

1. The first line of investigation by Reeves and coworkers involves the use of periodic acid. According to them, methanolyzed celluloses containing glycopyranoside end-groups would yield products retaining the methoxyl groups, whereas samples possessing open-chain dimethyl acetal end-groups would give rise to the half-acetal of glyoxal, CHOCH-(OCH_3)$_2$, and products devoid of methoxyl groups. Since the authors found that three samples of methanolyzed cotton linters, each containing 0.21% methoxyl, after having utilized 0.20, 0.46, and 0.83 mol HIO_4 per mol glucose anhydride showed no "significant" change in methoxyl, they reasoned that "had (the methoxyl groups) been combined as acetal all of the methoxyl should have disappeared in these experiments," and that "the complete removal of methoxyl . . . would have required only 0.034 millimol of periodate per g. (0.0055 mol periodate per mol glucose anhydride)." Obviously, this reasoning is based on the assumption that of the 182 glucose anhydride units (number average calculated on the basis of 0.21% methoxyl of dimethyl acetal origin), the terminal dimethyl acetal unit is the very *first* one which is destroyed by periodic acid. In the writer's opinion, such an assumption is just as unwarranted as the opposite view that this unit is the very *last* one attacked by the reagent. In the latter case no significant change in methoxyl would be expected until exactly 1 mol HIO_4 per mol glucose anhydride has been used. Actually, the change in methoxyl found by Reeves *et al.* (from 0.21% to 0.15%) amounts to 28.5% of the original value, and this drop could be readily interpreted as evidence for random removal of

a corresponding number of terminal dimethyl acetal units by periodic acid. However, for explanation of this "small" change the authors suggest "selective dissolution of the shortest chain molecules (those highest in end-group methoxyl content) after oxidation." Since distribution measurements [6] show that the methanolytic degradation products of celluloses are surprisingly homogeneous and do not contain unduly short chains, this explanation is not supported by known experimental facts. It is more likely that the observed 30% loss in weight of the 83% oxidized sample is caused by the increased solubility of those chains which become completely oxidized in the course of the topochemical reaction.

2. Reeves *et al.* base their argument on the fact that "fractionation of cellulose to approximately uniform molecular size would yield a sample having a number-average molecular weight only slightly lower than its weight average," and that "in no case could the number-average weight exceed the weight average." After "fractionation" of four different methanolyzed cotton celluloses with 10*N* sodium hydroxide solution at 0°C—a method hitherto unknown in the literature—they found 0.050%, 0.085%, 0.113%, and 0.18% methoxyl, and intrinsic viscosities corresponding to weight-average D.P.'s of 465, 296, 185, and 135 for the respective samples. From these data Reeves *et al.* concluded that "allowing 2 end-groups per molecule yields number averages (D.P. 760, 450, 338, and 212) greater than the weight averages," whereas "allowing but 1 end-group per molecule yields number averages (D.P. 380, 225, 169, and 106) slightly lower than the weight averages." Although the numerical values of the observed weight averages—on the assumption of virtually complete homogeneity—would allow the presence of 20% to 30% chains with dimethyl acetal end-groups and the balance with methyl glycopyranoside end-groups, the authors' interpretation may be challenged because it rests on the tacit assumption that the very small methoxyl values represent an absolute measure of the number-average molecular weights of cellulosic materials. Cellulose is not a pure chemical compound; it does not correspond to the ideal formula commonly used for its representation. Among others, it contains at least 0.04% to 0.052% carboxyl groups [12] and some 0.035% nitrogen [11], probably deriving from proteins which are chemically combined with terminal aldehyde groups. These foreign end-groups considerably diminish the number of free aldehyde groups in which, according to the ideal formula of cellulose, each and all of the chains are supposed to end. To be sure, in the methanolyzed samples the carboxyl groups should occur as —$COOCH_3$ end-groups. However, it has been shown by Hoffpauir and Reeves [3] that methoxyl groups of such nature are not measurable by their analytical procedure. While other legitimate corrections also could be applied in order to increase the methoxyl values which then would "prove" the presence of 2 methoxyl end-groups per molecule, the writer believes that the exceedingly small methoxyl contents, liable to corrections of 100% or more in the first two samples, do not represent a sound enough basis on which to support the claims made by Reeves and coworkers. Indeed, calculation of number averages from pertinent data given by them in Tables III and IV for another purpose yields results which—as is pointed out below—are inconsistent with their conclusion in this second line of investigation.

3. Since there are no chemical reactions known to distinguish between a glycosidic methoxyl and an acetal methoxyl, Mehta and Pacsu sought to obtain information as to the nature of the methoxyl groups present in the methanolytic degradation products of cellulose by studying the rate at which the reducing power of the samples increases during acidic hydrolysis. An alternative procedure for the rate measurement, adopted by Reeves and coworkers, consists in the determination of the methyl alcohol liberated from the hydrolyzed samples. This procedure was considered and rejected by Mehta and Pacsu for reasons discussed in the preceding paragraph. The hypoiodite method for estimating reducing end-groups was also discarded as an unsatisfactory procedure for polysaccharides [8]. Since it is known from the careful investigation by Birtwell, Clibbens, and Geake [1] that a quantitative relationship exists between relative viscosity and copper number over the most important range of the hydrocelluloses (between copper numbers 0.4 and 3.0), determination of the latter value would guarantee a precision equivalent to that of the viscosity measurements. Another obvious advantage is the consumption of comparatively large volumes of permanganate solutions from which the copper numbers are calculated. For these reasons Mehta and Pacsu adopted the copper-number method as a reliable and convenient means of studying the rate of restoration of the reducing end-groups in the methanolyzed samples. The results indicated

that the samples regained their reducing power much faster than they presumably would have if the blocking methoxyls had been of glycopyranoid nature. These experiments were repeated and the essential facts confirmed by Reeves and coworkers. However, they advanced the argument that "reducing action can be increased by the rupture of any (*sic*) linkage in the cellulose; therefore, increase in copper number should not be taken as evidence for the hydrolysis of methoxyl groups." In support of their assertion that "some hydrolysis occurs at positions other than the methyl glycoside groups," they refer to the D.P. estimations given in the last columns of Tables III and IV. Aside from the point that it is most unlikely that after the drastic acid treatment in the methanolytic process an incomparably milder acid treatment would cause new ruptures in the "limit hydrocellulose" chains, their D.P. measurements indicate an insignificant change only, amounting to -6%, which is well within the experimental error of viscosity measurements. It appears, therefore, that their argument is not substantiated by experimental evidence. The inconsistency in the experimental results of Reeves *et al.* becomes obvious on inspection of the data given in Tables III and IV. Whereas the weight-average D.P. (225) of the methanolyzed cotton cellulose sample in Table III is exactly the same as the number-average D.P. calculated from the methoxyl values (0.09%, 0.08%) on the basis of *1* methoxyl per chain, the weight-average D.P. (139) of the mercerized cotton cellulose sample in Table IV is greater than the number-average D.P. (120) calculated from the methoxyls (0.32%, 0.31%) even on the basis of *2* methoxyls per chain, which fact, by the argument advanced in their second line of investigation, would strongly indicate dimethyl acetal groups in the sample. Judging from the inordinately high copper number (2.3) and the very low methoxyl values (0.09% and 0.08%) of the sample in Table III, it seems almost certain that this material had already lost most of its methoxyls before it was subjected to the hydrolysis experiment. Similarly, the comparatively severe pretreatment amounting to "prehydrolysis" of the sample in Table IV should undoubtedly have resulted in a lower methoxyl content, and hence in an apparently lower value in the calculated rate of hydrolysis.

On the basis of the foregoing, the writer is of the opinion that the claim of Reeves *et al.* regarding the nature of the methoxyl groups in methanolyzed celluloses is not substantiated by their data. Instead, the presence of certain amounts of methyl glycosidic groups in the various samples remains a possibility, as was pointed out in the first part of this communication.

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EUGENE PACSU

Review of Textile Research and Development During 1950

by

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Review of Textile Research and Development During 1950*

by

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I. Summary

This paper is a review of the literature published during 1950 in the field of textile science and technology. The breadth of the subject matter in pure science and in technology represented by the extensive bibliography indicates the many important fields in which the textile industry is now devoting effort and which it must follow closely if improvements in processing operations and in materials are to be translated into more useful products for the consumer.

Papers in the field of research have been concerned with studies of the structure and properties of natural and synthetic fibers, with the chemistry of cellulose and starch, with the microbiological degradation of cellulose and wool, and with the processes of adsorption, swelling, diffusion, dyeing, and detergency. Certain general papers in the field of high polymers have been included because the techniques are important in textile research. Methods for determining molecular weight by viscometry, osmotic pressure, light scattering, and centrifugation have been reviewed. New equipments and techniques for the control of processing and laboratory equipment and for the test, identification, and examination of textile materials have been considered.

In the field of development, the papers in the literature have reported on improved processes for opening and blending, carding, drafting, spinning, winding, weaving, and knitting. In wet processing attention has been given to scouring, sizing, dyeing, and printing, bleaching, and stripping. The literature on finishing includes studies of dimensional stabilization of fabrics, flame-resistance, waterproofing, resin finishes, and coating materials.

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II. Introduction

The material reviewed in this paper has been selected from the scientific and technical journals published in the United States and abroad, and from certain standard abstract journals. It is believed that this review covers the important contributions in the fields of textile research and development during 1950; however, certain papers assembled at the outset have been omitted because they were considered inappropriate. The material in the text has been divided somewhat arbitrarily into the fields of research and development, and into sub-classifications of these divisions controlled principally by the published literature.

The bibliography has been arranged to conform with the main divisions and their sub-classifications. The individual papers cited are numbered consecutively in the bibliography; the papers in each of the principal sections in research and development are arranged in alphabetical order by first author. The numbers in brackets in the text refer to the appropriate papers in the bibliography.

III. Review Papers of General Interest

The general literature on textiles during the past year includes the publication of many books and scientific papers which are not concerned with particular materials or processes but deal in a broad way with the textile field. These publications treat such subjects as the general progress in research and development in the textile industry, general properties of textile materials, testing procedures, the dissemination of information, and the role of education with reference to the training of personnel for the textile industry.

An extensive list of the important books on textile technology and the textile industry, pub-

lished in English, has been compiled by Scott [31]. His bibliography is divided into twenty subject headings and includes a directory of publishers. A similar compilation has been prepared by Kertess [25]. Other general information publications include *Industrial Textiles Directory* [10], *Textile Catalogues and "Textile World" Yearbook 1949–1950* [3], *The Standard Handbook of Textiles* [19], *Dictionary of Textile Products* [20], and *Textile Raw Material Information* [22]. Karl [24] has considered textile materials from the point of view of their end-use in the rubber industry.

A report on the progress in textile research and development during 1949 has been published jointly by The Textile Institute and the Society of Dyers and Colourists [35]; a similar review has been prepared by the staffs of Textile Research Institute and The Textile Foundation [32], together with a classified listing of 826 references to the literature. Goldberg has summarized the achievements in the textile field in 1950 [17]. The current research programs of the Wool Industries' Research Association [4, 6], the Shirley Institute [9], Textile Research Institute [5, 12, 13], and the Danish Textile Research Institute [7] have been reviewed. Williams [36] has reported on the applications of science to industry and on what the research associations in Great Britain have accomplished. Schwarz [30] in a review of new developments in textiles has emphasized fields of study, such as the geometric structure of yarns, the end-use of products, and more precise statistical methods as fields where improvements may be expected. The functions of The Wool Bureau have become increasingly important in providing information concerning the wool research effort in the United States [21], in the exchange of research data on wool [1], and in suggesting research and development studies for the woolen and worsted industry [2].

In the educational field, Morton [28] has compared the systems of technical education for the textile industry in the United States with that for Great Britain. Fox [16] has given consideration to the role that research plays as an adjunct to the educational training of textile students. Draper [14] has made a detailed analysis of the recruiting and absorption of textile school students into the industry.

The general literature also contains a number of papers on studies relating to the fiber structure of both natural and synthetic textile materials. Studies of fiber structure have been reported by Brass [8], Dierkes [11], Elöd [15], MacArthur [26], Mönch [27], and Schams [29]. Webb [34] has made a comparison of the properties of various textile fibers. Illingworth [23] has published a review of recent periodical and patent literature on fibers and fabrics. Grove, Vodonik, and Casey [18] have reviewed the 1949–50 literature dealing with chemical treatments of natural and synthetic fibers, particularly treatments for mildew-prevention, fire-retardation, and water-repellency. Stoves [33] has pointed out the role of textile fibers in the field of archaeology.

IV. Research

A. Chemistry of Cellulose and Starch

1. Cellulose.—Hermans [59] has summarized in a monograph the results of his extensive research on the fundamental properties of cellulose, while Heuser [64] has discussed more briefly some trends in current cellulose research.

(*a*) Structure of Cellulose: Although Ant-Wuorinen [37], among others, has concluded that no carboxyl groups occur in carefully purified cellulose, Broda and Schönfeld [43] have interpreted the results of some experiments on the adsorption of radioactive ions by paper on the basis of carboxyl groups being the active centers. About one acid group per 125 glucose residues was found in modified celluloses. The possible occurrence in cellulose of acid-sensitive linkages, weaker than the glycosidic main chain bonds, is still unsettled. Cohen [47] reports some data on eucalypt cellulose which favor the theory of Schulz and Husemann, while Husemann and Goecke [68] have also made an examination of wood cellulose which supports this theory. A concept with several features of the theory of Pacsu has been put forth by Strepikheev [109]. Heuser [63], Hess [62], and Ward [114] have discussed morphological and structural factors in cellulose and how they can affect physical and chemical properties. Mühlethaler [85] has made some interesting electron microscope observations of the synthesis of cellulose by a bacterium.

Sippel [103] has suggested that the boat form for the glucose residues of the cellulose chains gives the best agreement with x-ray and physical data. McDonald and Beevers [78] have found that crystalline α-*D*-glucose has the chair form. Kiessig [73] has studied the alteration of the cellulose lattice by

water and found that the water molecules entering the lattice are statistically distributed, rather than being adsorbed on specific sites. Hermans, Vermaas, and Weidinger [60] have reported that mercerization of regenerated cellulose will increase the crystallinity observed by x-ray methods. Hermans and Weidinger [61] have also found that cellulose regenerated from very dilute viscose solutions is more crystalline than that from concentrated solutions. Battista [39] has investigated the recrystallization of ten representative celluloses during hydrolysis. The recrystallization is more pronounced for regenerated celluloses. Heath and Johnson [58] have studied the accessibility of wood pulp cellulose by the moisture-regain technique, and Stamm [107] has discussed the specific volume of native cellulose.

Hydrolysis studies have been extensively pursued as a means of investigating cellulose structure. Rånby and Ribi [91, 93, 94] have prepared peptized colloidal preparations of cellulose micelles, and have studied them by electron microscopy, x-ray and electron diffraction, and by other methods, particularly water vapor sorption [92]. The properties of these particles have been interpreted in terms of the structure of cellulose itself. Morehead [84] has also studied similar cellulose particles. Jørgensen [71, 72] has studied the differences in fine structure of natural cellulosic fibers by measuring chain-length distributions.

Investigations of cellulose and its derivatives by infrared spectroscopy [51, 75, 87, 99] have confirmed several previous conclusions about the structure of cellulose.

(*b*) Cellulose Solutions: Arkhipov and Kharitonova [38] have investigated the relation between ammonia and copper content of cuprammonium solutions and their ability to dissolve cellulose. Solution of the cellulose is believed to take place in two steps: (1) formation of a copper complex, and (2) solvation of the complex. A similar scheme has been proposed by Meyer, Studer, and van der Wyk [82] for the little-known phenomenon of solution of cellulose by organic solvents. Chloral-pyridine mixtures, for instance, are effective through formation of a cellulose-chloral complex, which dissolves in the pyridine. Borgin and Stamm [40, 41] have studied the solubility of cellulose in sodium zincate. Experiments with radioactive zinc indicate that the zinc complex is physically adsorbed rather than chemically reacted. The preparation of cellulose solutions for molecular-weight determinations is discussed in Section **IV**-I of this review.

(*c*) Cellulose Reactions: Stakheeva-Kaverzneva [106] has determined the ketonic carbonyl groups in a number of oxycelluloses. All of those oxycelluloses examined were found to contain some keto groups, except that prepared with periodate. Pinte and Rochas [89] have also indicated a method which will differentiate between ketonic and aldehydic reducing groups in degraded cellulose. Rogovin and coworkers have studied the preparation and properties of 6-carboxycellulose (prepared with NO_2) [97] and of dialdehyde cellulose and its esters [117]. Reeves and Darby [95] have shown that ethereal diazomethane or chlorous acid will stabilize dialdehyde cellulose—that is, cellulose oxidized with periodate—toward the solubilizing effect of alkali, but have suggested no mechanism for the stabilization. Head [57] has reported that light has a profound effect on the preparation of dialdehyde cellulose. Kleinert and Mössmer [74] have studied the mild oxidative degradation of cellulose with sodium toluenesulfonyl chloroamide and found two distinct reaction rates, which they interpret on the "weak link" theory. Davidson [49] has compared the affinities of a number of basic dyes for the carboxyl groups of acidic oxycelluloses. Haskins and Hogsed [55, 56] have begun a systematic investigation of the alkaline oxidation of cellulose with particular attention to the mechanism of the reaction. Sönnerskog [105] has proposed that ester resonance of the hydrogen atom on carbon atom number 1 of the glucose ring may be the mechanism of a number of reactions in the degradation of cellulose.

Jayme and Schenck [69] have applied their technique for measuring ease of acetylation to a number of wood celluloses and have shown how they rank in this respect. By streaming double-refraction measurements, Signer and coworkers [102] have shown that some cellulose esters are not molecularly dispersed in solution. The larger particles present are small crystallites, superficially esterified and solubilized. A similar phenomenon in aqueous solutions of carboxymethyl cellulose has been demonstrated by Dürig and Banderet [50]. Tattersfield [110] has investigated the effect of physical pretreatment on the ease of saponification of cellulose acetate, and has utilized the results to produce novel effects in fabric, to detect faults in yarns and fabrics, and to elucidate the surface structure of acetate

materials. Rabinovitch and Alexander [90] have begun an investigation of cellulose acetate hydrolysis by studying the alkaline hydrolysis of some sugar esters. Malm, Tanghe, and Laird [77] have studied the effect of a number of parameters on the percentage of primary hydroxyl groups produced in partially hydrolyzed cellulose acetate. Malm and coworkers [76] have also described the preparation and properties of pyridinium salts of cellulose acetate-chloroacetate.

Happey [54] has carried out x-ray and physical studies on the structure of cellulose and its derivatives; these studies have led to new approaches to the problems of acetylation, saponification, and nitration. The latter has also been treated by Chédin [45]. Rosenthal and Brown [98] have shown that the previous wetting history of linters can affect the degree to which they are nitrated. Jessup and Prosen [70] have estimated the heats of formation of cellulose and nitrocellulose, while Moore [83] and Nikitin [86] have studied the interaction of nitrocellulose molecules with solvents.

Studies of cellulose ethers have been primarily concerned with their use for studying the course of substitution reactions. Cohen and Haas [46] have studied hydroxyethyl cellulose and its preparation; Timell has studied methyl cellulose of low D.S. [112] and methyl and ethyl celluloses of higher D.S. [111]; and Rydholm [100] has studied the course of carboxymethylation. Brown and Thomson [44] and McLaughlin and Herbst [80] have reported on the preparation of sodium carboxymethyl cellulose; and Gloor, Mahlman, and Ullrich [53] have discussed hydroxyethyl cellulose and its uses.

Heuser, Heath, and Shockley [65] have found that the rate constant for tosylation of primary cellulose hydroxyls is 5.8 times that for the secondary groups. Hoffpauir and Guthrie [66] have examined the ion-exchange properties of a number of modified celluloses. Breslow [42] has prepared some acid-soluble cellulose derivatives, a typical one being ethyl cellulose β-pyridyl carbamate. Woiwod [116] has made the interesting observation that the fluorescence of amino acids, peptides, and amines on filter-paper chromatograms is due apparently to a reaction between aldehydic groups in the paper and amino groups in the chromatographed compound. Reeves, Masseno, and Hoffpauir [96] have methanolyzed native and regenerated celluloses and suggest that the course of the reaction is determined by a coalescence of crystalline areas when internal stresses are relaxed by the chain-splitting of the reaction. Sohn [104] has studied the formation of reductic acid by oxalic acid hydrolysis of various celluloses.

(*d*) Analytical Procedures: Windeck-Schulze and coworkers [115] have critically evaluated the dichromate oxidation technique for determining cellulose and suggest improvements. Viles and Silverman [113] described the use of anthrone as a colorimetric reagent for cellulose and starch. Meller [81] and Husemann and Consbruch [67] have found potassium permanganate unsatisfactory in end-group determinations, and Salova [101] has adapted the Rehberg method to the microanalysis of uronic acids in cellulose. Genung [52] has given a general discussion of problems in the analysis of cellulose derivatives. Three methods for determining the degree of substitution of carboxymethyl cellulose have been published [48, 79, 88]. Kuhn [75] has suggested an infrared spectroscopic technique for determining the degree of nitration in cellulose. Stark [108] has proposed a colorimetric determination of pectin in cotton.

2. Starch.—

(*a*) Structure of Starch: Kerr [140] has published a second edition of his comprehensive monograph on starch, and Meyer and Bernfeld [146] have prepared a less extensive review of the chemistry of this important material. Lipatov, Kartsovnik, and Shul'man [141] have studied the structure of potato starch in variously prepared solutions by means of measurements of the dielectric constant, heat of solution, osmotic pressure, and several other properties. Kerr [139] has found that the oxidation of starch with nitrogen dioxide confirms qualitatively the fact that amylopectin is more extensively branched on carbon atom 6 than amylose. Wolfrom and coworkers [158] have disclosed the isolation of crystalline isomaltose octaacetate from amylopectin after acid degradation and acetylation. Baker and Whelan [118] reported that the birefringent characteristics of structural starches are due more to differences in orientation than to differences in relative proportions of amylose and amylopectin.

(*b*) Enzymatic Hydrolysis: The mechanism of the hydrolysis of starch by β-amylase is still in question. Kerr [138] has presented evidence in favor of the "straight chain" theory, while Hopkins and Jelinek [134] and Bourne and Whelan [122] have supported

the "multi-chain" theory. Recent developments in the field of amylase chemistry have been reviewed by Redfern [151]. Lalla [144] has observed an increase in the viscosity of starch in the initial attack of a bacterial amylase on starch. Hopkins and Jha [135] have studied the hydrolytic action of β-amylase on amylose and amylopectin, and Hirst and coworkers [131] have compared the action of β-amylase on amylopectin and on glycogen. Barker, Bourne, Wilkinson, and Peat [119] have reported on the properties of purified *P*-enzyme and *Q*-enzyme, and Gilbert and Patrick [129] have reported that the latter compound has been crystallized. Barker and coworkers [120] have apparently proven that the *Q*-enzyme works by a non-phosphorolytic mechanism.

(*c*) Starch Derivatives: Roach and Nordgren [152] have reviewed the properties of allyl starch and its use as a polymerizable film-forming resin. Two methods have described the preparation of mixed allyl ethers. In the first method [123], the allyl ether group was introduced at the same time as, or subsequent to, an alkyl or acyl ether group; while in the second [153], an acyl group was added to the allyl starch ether. Israelashvili [136] has proposed a mechanism for the nitration of starch. Lohmar and Rist [142] have discussed the acetylation of starch. Lohmar, Sloan, and Rist [143] have studied the phosphorylation of starch and have shown how the reaction conditions determine the character of the product. Katzbeck and Kerr [137] and Dvonch, Yearian, and Whistler [124] have investigated the complexes which amylose forms with organic substances and iodine. The degree of polymerization and the molecular configuration appear to be important factors in their formation. Haller [130] has characterized some of the familiar complexes formed by starch and metal salts. Möller [147] has discussed some of the uses of starch ethers in the textile industry.

Norberg and French [150] have studied the redistribution reactions of macerans amylase with Schardinger dextrins and the action of soybean β-amylase on amyloheptaose [126]. Myrbäck and Järnström [148, 149] have studied the acid hydrolysis and periodate oxidation of Schardinger dextrins. French and McIntire [127] have oxidized the α-, β-, and γ-dextrins with periodate, and found that they produce no formic acid or formaldehyde. French, Knapp, and Pazur [125] and Freudenberg and Cramer [128] have both concluded the γ-dextrin is the ring homolog of the α- and β-dextrins and is cycloöctaamylose.

(*d*) Miscellaneous: Whelan and Peat [155] have studied the photochemical degradation of starch and found that two distinct types of degradation occur, depending on whether oxygen is present during the reaction. McCready and coworkers [145] have described a method for determining starch and amylose in vegetables, and Wolff, Gundrum, and Rist [157] have described a simplified procedure for the characterization of starch fractions. Hofstee [133] has found that the concentration of starch pastes affects the cooking time required to reach maximum viscosity in a regular manner. He has also studied [132] the action of HCl on potato starch and the effect this has on gel formation. de Willigen [156] has investigated the influence of small concentrations of electrolytes on the viscosity and gel strength of potato starch solutions, and Bechtel [121] has described a gelometer for measuring the properties of starch gels. Ulmann [154] has chromatographed soluble starch on a column of alumina and has separated it into three fractions which can be identified by the colors they form with iodine.

B. Structure and Properties of Cellulose Fibers

1. Bast Fibers.—The principle problem in the production and utilization of bast fibers is that of decortication. It is therefore appropriate that this problem should be given the most attention in the literature [200]. Several methods of decortication of bast fibers by using water and steam [183] and bacterial enzymes [163] have been reviewed.

The importance of jute among the bast fibers is emphasized by the number of reported investigations on this fiber. Studies have been reported on its chemical composition [169, 173, 195], structure [196, 197], mercerization [188, 189], frictional properties [161], bleaching [168, 185, 187], and photochemical discoloration caused by urea-formaldehyde treatment [170]. The decortication of jute is commercially accomplished by the use of bacterial pectinases with softening agents [162]. The latter are added to reduce the rigidity of jute fibers so that they may be spun readily [171, 172]. The swelling of jute in bales has also been measured [177].

Flax varieties have been studied both chemically [190] and histologically [199]. This fiber may be decorticated or retted by bacterial means [182] both aerobically [166, 176] and anaerobically [167, 198],

by chemical delignification [165], and by combination mechanical and chemical continuous process [191]. The method of retting has an important influence on the quality of the resulting linen fibers. Treatment with an emulsion of fats and mineral oils softens the fibers and improves their spinning properties [186].

Several reviews [179, 180, 184] reflect the continued interest in ramie as a potential native cellulose fiber. Drying data have been obtained for ramie [178, 194] which will be useful in processing operations. A method for determining the fineness of ramie fibers by fluidity measurement has been proposed [160].

Reports have appeared in the literature on the fine structure of sisal [192], the quality of abaca [164], and the chemical composition of Manila hemp [159].

The demand for hard fibers and hard-fiber products [181] has called attention to less common sources of raw materials. Among those being considered is yucca [174], kenaf [193] as a substitute for jute, and nettle [175].

2. Cotton.—Additional results of chemical analyses of cotton fibers have been reported. Trace metals were determined by means of spectrochemical analysis [208] and the presence of galacto-araban has been demonstrated in the raw cotton [207]. The wax contents determined for 62 samples of American upland cotton were found to be closely proportional to the surface area per unit weight of fiber [217]. The use of wax-content measurement has been proposed as a method of evaluating the maturity of a cotton sample [212, 218].

Pearson has made extensive measurements of the perimeters of cotton fibers in the primary-wall stage [220]. Goring and coworkers [204–206] have begun an intensive investigation of the electrokinetic properties of cotton and other cellulose fibers. They believe that the zeta-potential observed for cotton in the form of pads is related to the swelling of the fiber but have so far been unable to account for their observations. Wetting characteristics have also been studied [221].

Kast [215] has reviewed the x-ray structure of cellulose fibers and has attempted to explain their properties in terms of structural components: elementary crystalline cell, crystallites, transition regions, and noncrystalline areas. Heyn [211] has attempted to evaluate crystallite size by means of small-angle x-ray scattering. Density measurements are still being used for fine-structure studies [209, 210]. Cotton fibers ground in a fibratory ball mill for suspension in an infrared absorption study were found to be converted completely into amorphous cellulose [51].

An interesting comparison of the mechanical properties of cotton and rayon fibers made some years ago in the Danish Institute for Textile Research has recently appeared in the literature [201, 202]. An electrical apparatus for measuring the extension and force imposed on single cotton fibers during longitudinal vibrations has been developed. With the aid of this apparatus, studies of the dynamic moduli, stress relaxation, and fatigue were made.

Investigations using x-ray, chemical reactivity, and electron microscope techniques indicate that chemical or mechanical breakdown of cotton fibers occurs initially by degradation of the cellulose between the fibrils in the fiber [213]. This fact is the basis of a test of injury to cotton fibers which depends upon the solubility of exposed cellulose fragments in caustic soda solution [226]. The effects of alkali silicates on the fibers can be seen microscopically [223].

Treatment of cotton linters with silicon tetrachloride, hydrolysis, and burning-out of the organic portion of the fibers left silica pseudomorphs of the original fibers with fibrillar structure [216]. This technique would seem to bear further investigation for structure studies.

Further investigation of a test utilizing iodine sorption as an indicator of mercerizing defects has lead to the conclusion that the iodine sorption is a measure of the amorphous fraction in the fiber [224, 225].

The Goldthwait differential dye test has been applied to a study of structural changes in the cotton fiber as it dries in the boll [203] and to the evaluation of the suitability of linters for viscose manufacture [219].

Rutherford has compared the dyeing characteristics of irrigated and rain-grown cottons [222]. It has been shown that abrasion of the fiber increases its dye absorption [214]. Further work on the use of ethyleneimine and similar reagents to permit the use of wool dyes on cotton has been reviewed by Wigman [227].

3. Rayon.—This section is concerned only with the properties and structure of rayon filaments and does not deal with problems in connection with the utiliza-

tion of pulps or linters or processes for the production of the fibers by the chemical industry.

One of the most fruitful approaches to the structure of rayon fibers has been through the medium of x-ray techniques [244]. Kast and coworkers, beginning in 1948, have published a series of papers on the x-ray structure of rayons as modified by mechanical stretching during the spinning process [234–236]. The small-angle scattering of rayon filaments has also been investigated [233]. The crystallinity of such fibers was determined from the measurement of the relative proportions of the incident x-ray beam scattered into the diffraction peaks and into the background [228]. X-ray diagrams have also been used to follow the acetylation and subsequent hydrolysis of the acetylated cellulose fibers [230].

Recent work has shown that the viscose filaments are fairly heterogeneous in structure. It has been known for some time that rayons have skins of crystallinity and orientation which differ from the core of the fiber. Hermans [231] has demonstrated these features with the electron microscope and, using a peeling-off method first demonstrated by Elöd, has measured the difference in reaction rates and structure of the core from that of the skin [232]. The skin is removed in successive layers by acid-catalyzed topochemical acetylation and subsequent solution of the acetylated shell. Swelling, birefringence, and electron-microscope studies were made as various amounts of the fiber were removed for several types of rayon, and a detailed picture of the internal structure of the fibers was obtained. Similar electron-microscope studies have been reported by Mühlethaler [238], who observed changes in the fine structure of viscose rayon during solution and regeneration.

The recent literature also contains reports of studies on the conductivity of viscose films impregnated with various salt solutions [239], the abrasion of single rayon filaments [229], the effects of wet-stretching treatments [240], and the changes produced by various drying methods [237, 243]. The effects of bleaching and steaming treatments on lustrous and delustered viscose rayon with respect to degradation and dye affinities were reported [241]. Results of research conducted in Russian-controlled Europe on viscose rayon shrinkage have recently been published [242].

C. Structure and Properties of Protein Fibers

1. Structure and Properties of Wool.—The molecular structure of keratin fibers, together with the relationship of structure to the observed physical and chemical properties, received wide attention in 1950. Stoves [304] has reviewed the literature bearing on the structure of wool and on physical and chemical processing. The structure of wool fibers has also been discussed by Lindley [283], Goodings [263], and Alexander and Earland [245]. The application of x-ray diffraction techniques to the study of keratin and other proteins has been reported by Kratky [273], Nutting [296], Hodgkin [268], and Bear and Bolduan [252–254]. Bear and Bolduan have given a detailed description of the equipment which they have developed to take x-ray diffraction photographs of long periodic spacings (800 Å. to 1400 Å.) for studies of protein-fiber structure. Bond lengths and bond angles of the polypeptide chain of proteins have been derived by Corey and Donohue [260] through the analysis of data from recent crystal-structure studies of amino acids and peptides. Bragg, Kendrew, and Perutz [255] have conducted a systematic survey of polypeptide chain configurations which conform to established bond lengths and angles and are held in a folded form by N—H—O bonds. The results of Perutz and Kendrew with crystalline haemoglobin and myoglobin agree with Astbury's studies on α-keratin in indicating the existence of a folded polypeptide chain with a repeat distance of about 5.1 Å. and with three amino-acid residues in this distance. The two-dimensional Patterson projection of a plane at right angles to the chain appears to favor the general type of structure for α-keratin proposed by Astbury, and gives good agreement between calculated and observed F values when these chains are placed in the myoglobin structure and account is taken of the side-group scattering. The comparison with haemoglobin is not at all close; this leads the authors to state that extreme caution must be exercised in structure comparisons of this type and that at present the evidence is too slender for conclusions to be drawn. Pauling and Corey [300], in connection with their research program for determining the detailed atomic arrangements of crystals of amino acids, peptides, and other simple substances related to proteins, have constructed two hydrogen-bonded spiral configurations of the polypeptide chain, with residues all equivalent except for variation in the side-chain. One of these spirals is a three-residue spiral, in which there are about 3.7 residues per turn, and each residue is hydrogen-bonded to the third residue from it in each direction along the chain, giving a unit transla-

tion per residue of 1.47 Å. The authors point out that there is evidence that this configuration is present in α-keratin, contracted myosin, and some other fibrous proteins, and also in haemoglobin and other globular proteins. The second hydrogen-bonded spiral is the five-residue spiral, with about 5.1 residues per turn, each residue being bonded to the fifth residue from it in each direction. The unit translation is 0.96 Å. The authors believe that this spiral is present in supercontracted keratin, which is formed from α-keratin with a shrinkage of about 35% in the fiber direction. Mizushima [292] has proposed a theory to account for the intramolecular-rotation potential forming coiled or straight peptide chains. Mazia [289] has concluded that some birefringent protein fibers are not composed of fine fibrils but are highly folded thin sheets.

Uzman and Blout [306], in a study of films cast from native, regenerated, and denatured pepsin solutions, have reported an increase in the absorption of the infrared band for denatured pepsin at 3260 cm.$^{-1}$ (3.07 μ); this band is attributed by Sutherland and his coworkers to a hydrogen-bonded N—H frequency. These results have been interpreted to mean that a greater number of hydrogens associated with nitrogen are hydrogen-bonded to neighboring atoms in the denatured state. Lenormant [280] has studied the infrared spectra of proteins in the 6 μ region and has emphasized the importance of the active form of the N—H bond, which gives an absorption band at 6.4 μ, as compared with the simpler structure which absorbs radiation at 6.0 μ. The 6.4μ band appears to be due to a special state of the amide group in which there is an internal rearrangement of the linkages tending to a symmetrical division of the double bond, associated with a perturbation of the N—H bond, giving the hydrogen a much greater activity. Mizushima and coworkers [294] have concluded that the peptide bonds in the main chain of polypeptides cannot be considered to be in the *cis*-configuration, based on the presence of the infrared absorption bands observed in the regions 3210–3240 cm.$^{-1}$ and 3070–3100 cm.$^{-1}$. These authors have pointed out that their conclusions are at variance with the results obtained by Darmond and Sutherland but are in support of the type of molecular structure proposed by the authors for α-keratin [293]. Haurowitz [266] has conducted studies on the structure of spheroprotein molecules and claims that they are held together primarily by salt bridges, also by dithio linkages and by the branching of peptide chains, and possibly by the interaction of groups in the component amino acids, such as —COOH, —NH_2, —SH, or —OH.

The relation of the chemical structure of wool and hair to the observed physical properties has been a field of active investigation. Ripa and Speakman [303] have reported that the sulfur content of wool fibers may be closely related to their creep characteristics in water. Reese and Eyring [302] and Katz and Tobolsky [271] have studied the stress-relaxation of single wool and hair fibers, respectively, in various aqueous solutions. In effect, these studies make use of the rate of relaxation of stress to provide information about the rate of chemical reaction between the stress-supporting bonds of the fiber and the reacting solution. The reaction of various chemical agents and hair fibers was found by Reese and Eyring to be first-order with respect to the reagent and the number of bonds to be broken in the fiber. The retarding effect of the diffusion of a reactant into the fiber has been analyzed by these authors and by Katz, Kubu, and Wakelin [270]. Burte has used a low-elongation mechanical technique to study changes in the Hookean slope of single wool fibers in concentrated [258] and in dilute neutral salt solutions [257].

The extent to which a keratin fiber supercontracts following a thermal or chemical treatment has been used as an indication of structural modification to both wool and hair fibers. Fearnley and Speakman [262] have described the use of this technique, after treatment of wool with di-epoxides, in order to study the effect of the cross-linkage formation between molecular chains. Brown, Pendergrass, and Harris [259] have studied the supercontraction in bisulfite of fibers modified with hydrosulfite and ethylene dibromide and have shown that the modified fibers contracted less than 1% while the untreated fibers supercontracted 20%. Zahn [311, 312] has observed supercontraction of horse-hair of more than 30% after heating the fibers in purified phenol; he has also determined the influence of temperature on the contraction of keratin fibers in a 50% phenol solution [310] and has studied the effect of heat on the relaxation of prestretched wool [308]. Elöd and Zahn [261] have measured the thermal expansion coefficient of keratin fibers and have found that this coefficient is positive for normal fibers, either wet or dry. After treatment with phenol and following the subsequent supercontraction, the expansion coefficient becomes negative. The authors conclude that

this is evidence for two different structural states for keratin fibers—one a normal solid with positive coefficient of expansion and high elastic modulus, and the other a partial liquid body with negative coefficient of expansion and low modulus.

The status of work on the surface structure of the wool fiber has been reviewed by Lindberg [281], Mercer, Lindberg, and Philip [291], and Zahn [309, 313]. Appleyard and Greville [250] have used ultraviolet light to study the longitudinal sections of human hair and Lincoln and Leicester wool fibers; they found the scale thickness of hair to be about 0.33μ, while that of the wool fibers studied was 0.66μ. The cuticle of the wool fiber was estimated from both transverse and longitudinal sections to be much thinner than that of human hair, hog bristle, or alpaca. This difference in cuticle thickness is probably due to the different arrangement of the scales in wool and in hair.

Replica techniques have been found useful in studying the structure of the surface and the cuticle cells of the wool fiber [287]. Mercer [290] has published evidence that the so-called "subcuticle" in wool and hair overlies the scale structure. He has coated the outside surface of the fiber with gold metal and has separated the thin-walled membranes by treating them with phenol and digesting with trypsin. There appears to be some question, however, concerning the interpretation of this work [307]. Lagermalm and Philip [274] have described a short-time treatment for isolating the epicuticle from wool by the use of dilute Na_2S or KOH. Alexander and Earland [246] have reported that the treatment of wool with peracetic acid may be used as a method for separating a layer, probably identified as the "sub-cutis" from the wool fiber.

Olofsson [297] has made a theoretical study of fiber friction, and Olofsson and Gralén [298] have surveyed the work on the measurement of the frictional properties of wool and rayon staple fibers. King [272] has measured the frictional coefficients of wool by a slip-stick technique against such materials as horn, nylon, and ebonite; coefficients of friction for nylon fibers against a nylon surface were also measured. For wool both the with-scale and anti-scale frictional coefficients increased with moisture regain. The frictional coefficients for nylon behaved similarly. The action of swelling agents on the frictional coefficients of wool and nylon indicate that a close relationship may exist between the elasticity of the rubbing materials and their coefficients of friction. In commenting on King's work, Makinson [286] has pointed out that, although the frictional behavior of the surfaces must be related to their elastic and plastic properties, there is no reason why such a relationship should be simple. Makinson has repeated some of King's experiments and finds that the friction increases with the change from a saturated atmosphere to immersion in liquid water, while King has observed a decrease for both wool and nylon. It has been pointed out by Gralén [264] and Lindberg and Gralén [282] that the low value of with-scale frictional coefficient for wool (0.15) may be explained by the fact that the wool fiber is covered by a thin membrane, the epicuticle, which has a low chemical reactivity.

Hopkins [269] has reviewed those chemical and physical properties of wool which are important for its use as an apparel fiber. Factors affecting the growth of the wool fiber [249, 277], such as the dimensions of follicles and other structures in the skin of sheep [256], the environment [265], and diet [299], have been discussed in the recent literature. The relation between strength and diameter of single wool fibers selected from the fleece of a one-year-old Romney lamb has been studied by Anderson and Cox [247]. Based on force-elongation measurements of 185 single wool fiber samples, each 1.5 cm. long, the authors have reached the following conclusions: "(1) fibre breaking load per unit cross-section increased with fibre diameter; (2) fibre breaking load was positively correlated (for fixed diameter) with fibre breaking extension; (3) there was a small positive correlation between fibre breaking extension and fibre diameter; and (4) the percentage coefficient of variation of fibre diameter within 1.5 cm. lengths was 10.3 ± 4.25."

Length and fineness measurements and testing procedures have been reported [267, 279, 288, 295, 305], as well as the presence and effect on processing of non-kempy medullated fibers of Australian wool [275]. Lang [276] has reviewed methods of testing raw wool and has discussed various techniques for determining moisture content, yield, fineness, and fiber length of raw-wool lots. Lipson has discussed various chemical methods of testing which are applied to the preliminary stages of processing, such as wool scouring [284], and to the later stages of manufacture, including dyeing and finishing operations [285]. The measurement and significance of crimp

as a physical parameter have been reviewed [248, 278], and an attempt has been made to relate the length of the wool follicle to the crimp characteristics of fibers [251]. There appears to be some correlation for Merino wools but not for the Lincoln wools studied. The introduction of methods for the artificial crimping of fibers has been shown to induce profound changes in their processing behavior and in the quality of finished materials fabricated from such crimped fibers [301].

2. Chemistry of Wool.—Literature on the chemistry of wool covers a wide range of subjects dealing with such matters as the chlorination of wool, the action of acids, bases, and oxidizing agents on wool, methods for estimating damage to wool, and analytical procedures for end-group determination. A brief summary of the work of Elöd, Schöberl, and the I.G. Farbenindustrie A.-G. on wool has been presented by Evans [330]. An excellent summary and review of the chemistry of wool has been presented in the *Wool Science Review* [322]; general articles of this kind have also been prepared by Mönch [346] and Soper [351]. Zahn [356] has studied the general problem of the changes in wool caused by water over a wide range of temperatures and times, the hydrolysis of wool with HCl, and the effects of dry heat in the range 120°–210°C. Under the latter condition, slow carbonization takes place, and esters of cystine, arginine, proline, and threonine are separated.

The action of acids and bases on protein fibers has been considered by Fröhlich [332]; Steinhardt and Zaiser [354] have studied the combination of wool protein with cations and hydroxyl ions. Titration curves for wool at 0°C with 12 strong univalent bases show the same wide differences with respect to their position on the pH axis as those found with acids. Similar to the anion behavior, the affinity of cations for proteins increases with molecular dimensions. The explanation of the degree of shift of the titration along the pH axis, and the selective effect on the dissociation of lysine are suggested by these authors. Elöd and Fröhlich [328, 333] have presented data on the absorption of NaOH by wool and silk, indicating that they behave similarly in the region between pH 7 and pH 13.5. Factors concerned with the acid sensitivity of wool and its isoelectric point have been briefly discussed [321].

Lindberg [339] has measured the rate of acid diffusion into wool fibers by using a conductivity cell and measuring the decrease of bath concentration with time. He has emphasized the importance of the epicuticule in the diffusion process; the presence of an undamaged epicuticle gives a low diffusion constant.

The chlorination process for wool has been reviewed [323]. Published studies have indicated that, although sodium chlorite should not be used as a bleaching agent because of its degrading effect on wool, it may be used under well-defined operating conditions to impart a certain degree of resistance to shrinking [348]. Alexander, Gough, and Hudson [319] have studied the kinetics of the reaction of wool with chlorine solutions and have found that the rate is always proportional to the initial concentration and that diffusion proceeds from a changing surface concentration.

Das and Speakman [326] have reported that when wool is treated with 1.25*N* solution of chlorine dioxide in the absence of light at 22°C, there is a rapid drop in cystine content, about a quarter of the sulfur in the wool being oxidized ultimately to sulfuric acid and the remainder to cysteic acid. After a treatment of 120 hrs., the reaction is essentially complete, and about 40% of the protein is dissolved during the treatment and subsequent washing. By studying the difference between the complete oxidation of cystine in wool by Cl_2, HClO, or peracetic acid, and the partial oxidation of cystine by permanganate or alkali hypochlorite solutions, Alexander, Hudson, and Fox [320] have concluded that wool must contain two fractions of cystine with different reactivities. As an explanation, the authors have suggested that the reactivity of the disulfide group depends strongly on induced electric effects arising from neighboring groups. Studies of wool in reaction with hydrogen peroxide [317] have shown that the latter is held by both the amino and carbimino groups and that the sorption process resembles the formation of complexes with simple organic nitrogen-containing compounds, such as urea. Fabrics of wool are found to be made unshrinkable by less than 10% adsorption of the condensate from di-*n*-butyl ether of dimethylolurea; this treatment also gives the fabric greater abrasion-resistance, and yarns so treated have an increased tensile strength [316]. Zahn [357] has experimented with stabilizing wool and silk with 1,3-difluoro-4,6-dinitrobenzene and has reported that in these reactions the lysine side-groups are mainly involved. Alexander, Carter, and Earland [318] have found that treatment of wool with chlorosulphamic

acid renders the wool completely unshrinkable, provided it is used with high concentrations of sodium chloride. Alexander, Bailey, and Carter [315] have studied the application to wool of *N*-carboxy anhydrides of amino acids for the control of felting shrinkage. From studies of the reaction of lithium bromide on wool, Alexander [314] has suggested that fibrils are formed from micelles held together by cystine bonds and that micelles are formed from macromolecules through hydrogen bonds and a few cystine bonds.

Ellis, Gillespie, and Lindley [327] have conducted biochemical studies of the wool root; aqueous or 70% ethyl alcohol extracts of this material were analyzed for amino acids by paper chromatography, and glutamic acid, glycine, alanine, valine, leucine, and serine were identified conclusively. Wool roots contained about 2.3% sulfur, of which 0.7% was present as —SH and 1.55% as —S—S—. Dipeptides of cysteic acid with nine amino acids have been identified chromatographically in oxidized partial hydrolyzates of wool [325]. Middlebrook [345] has described the use of 2,4-dinitrophenyl derivatives to identify end amino groups in wool. Spies and Chambers [353] have reported on the effect of alkaline hydrolyzing agents on free and peptide-linked tryptophan. Ingram [336] has described a micro end-group method for peptides. The action of potassium chloride on keratin [352] and of chloromethyl ethers on wool for washing-resistance [337] have been reported. Attention has also been given to the formation of polymers in wool [331, 340]. Sobue [349, 350] has reported on the results of his study of reactions of sulfuryl chloride with keratin fibers. Products of the hydrolysis of wool keratin by papain [324] and photolysis of the peptide bond at 2537 Å. have been discussed [341].

Tests for damage to wool have been reviewed by Elsworth [329], Glynn [335], Kornreich [338], Genet [334], and by Meeuse, Hietink, and Gorter [342]. The last is an extensive review of the causes and treatment of damage in wool, as well as a tabulation of methods for testing wool for damage. This survey contains 292 references to the published literature. van Overbeke, Lefevre, and Raux [347] have used alkali solubility for determining wool modification. Zahn [355] has tested methods for detecting damage in wool, including alkali solubility, dyeing tests, and supercontraction with samples of wool pretreated with various alkalies and acids. He has concluded that the causes of wool damage cannot easily be found by one method but must be established by a combination of a number of test methods. One of the methods proposed by Zahn is the interesting possibility of using supercontraction of wool yarns as a measure of determining the alkali damage. Menkart and Mitchell [343] have used this technique and have measured the supercontraction of yarns and single fibers removed from the yarns. The yarns were treated with sodium metabisulfite, following various alkali pretreatments in the temperature range 25°–80°C. Their measurements indicate that the supercontraction effects observed in yarn do not give a dependable estimate of the supercontraction characteristics of its constituent fibers. Mercer and Roadknight [344], using optical and electron microscope techniques, have examined the effect of chlorine, enzymes, and alkalies on damage to the fiber. This type of damage does not necessarily destroy the fiber and may provide some desirable characteristics, such as shrink-resistance.

3. Structure and Properties of Silk.—Several publications on silk technology and classification [374] and on the German [372] and Indian [360] silk industry provide general background on methods used in the processing of silk.

Kratky, Schauenstein, and Sekora [364–366] have made a study of the structure of silk fibroin and have found that the native silk fibroin becomes highly crystalline on drying. The extremely sharp x-ray diffraction patterns from the dry silk fibroin suggest that the crystals form without interference and appear to be much larger than those of the spun silk fibroin. These authors have suggested the names "Silk I" for the structure which the air-dried native silk fibroin presents, and "Silk II," which is the well-known x-ray pattern produced by the cocoon filament which may be obtained from the air-dried fibroin by mechanical treatment of the sample. If "Silk II" is treated with 0.5% NaOH at 60°–70°C for about a minute, side-chain spacings of 45 Å. appear. This crystalline form of silk has been called "Silk III." "Silk III" shows all of the interferences produced by "Silk II" with the following additional spacings present: 8.9, 11, 15, 23, 31, and 45 Å. These authors have commented on the earlier work of Coleman and Howitt, who have cast films of denatured silk fibroin dissolved originally in cupriethylenediamine and have studied the properties and structure of these films. Coleman and Howitt found that

these films were crystalline if dried rapidly. Kratky and coworkers point out that if such a solution is dried slowly, "Silk I" will be formed; while if it is dried rapidly, the normal β x-ray pattern of "Silk II" will be formed. They point out that since Coleman and Howitt were able to identify a globular form of the protein in their solutions, the opinion is confirmed that "Silk I" obtained by very slowly drying the cupriethylenediamine solution of silk fibroin represents a globular protein. Drucker and Smith [359] have also dissolved silk fibroin in cupriethylenediamine solutions. After neutralizing the solution and dialyzing, it was found that part of the silk fibroin precipitated out when the resulting aqueous solution was adjusted to a pH of 3.0. The precipitate, the soluble fibroin, and the original fibroin were hydrolyzed with HCl and the residue studied by using paper chromatography. Each component gave identical chromatograms with regard to position, size, and intensity of amino acid spots. The presence of aspartic acid, glutamic acid, serine, threonine, glycine, alanine, valine, leucine, phenylalanine, tyrosine, arginine, and proline was demonstrated. A 30-min. tryptic hydrolysis of the soluble fibroin produced a precipitate consisting entirely of glycine, alanine, and serine, with the molecular weight 7000 based on viscosity measurements of a cupriethylenediamine solution. Drucker and Smith have proposed a possible structure of the folded molecule of denatured fibroin based on these experiments. In the fibroin molecule of molecular weight 33,000, they proposed a structure containing three straight-chain sections with a total molecular weight of 7000 each and containing only glycine, alanine, and serine linked together by two regions of molecular weight 5000 each, which contain all the bulky amino acids of the molecule. The authors also point out that such a structure is comparable with the x-ray results obtained by Myer, Fuld, and Klemm, who have showed that the fibroin has an x-ray pattern which indicates a crystalline peptide chain consisting only of glycine, alternating with alanine (or serine). By treatment of the fibroin with diazobenzenesulphonic acid (Pauley reagent) which combines with tyrosine, it was found that the x-ray diffraction pattern of the fibroin was unchanged, showing that tyrosine is probably located in the amorphous region of this polymer. Rockland and Dunn [371], by the use of a direct photometric analysis of color intensities of filter paper chromatograms stained with ninhydrin, have determined that silk fibroin contains 43.4% glycine and 34.9% alanine. Levy and Slobodiansky [367] have used the isotopic derivative technique to analyze partial hydrolysates of silk fibroin during various stages of hydrolysis. They have given particular attention to the amounts of alanine, glycine, glycylalanine, alanylglycine, and glycylglycine which are produced at various stages of hydrolysis. They find that the liberation of free alanine and glycine follows the kinetics of a first-order reaction.

An analysis of silk fiber [370] from Italy, France, Japan, Iran, and various parts of China showed small but significant differences in the contents of glycine, alanine, serine, and tyrosine, and in the glycine, serine, and tyrosine of sericin. Zahn and Kohler [376] have studied silk which was heated with 1.5*N* HNO_3 at 30°, 43°, and 70°C. On washing and air-drying, it was found that the degree of proteolysis increased with the temperature; the three preparations contained 2.7%, 1.4%, and 0% of tyrosine, respectively. When silk was nitrated at 43°C, the x-ray diffraction patterns showed the development of long spacings (36–40 Å.). It was also found that when human hair containing 13.2% cystine and 0% tyrosine was heated with 1.0*N* HNO_3 at 70°C for 24 hrs., x-ray diffraction patterns showed long spacings both at 40 Å. and at 83 Å. Coulombre and Moore [358] have measured the adsorption of sulfanilamide on silk fibroin at a pH of 6.0 at 0.1°, 16.3°, and 34.4°C, and have found that the differential heat of adsorption remains constant at 4500 cals. per mole until an adsorption of 0.0058 moles of sulfanilamide on 100 g. of proteins was reached. After this, the rate decreased rapidly. This rate is probably connected with lysine side-chains in the fibroin, whose combined concentrations total 0.0061 moles on 100 g. of protein. Goldstein and Halford [361] have made a preliminary report of the examination of silkworm gut which shows regions of high organization under polarized infrared radiation. Yamafuji [375] has fed silkworms mulberry leaves which were moistened with both potassium and sodium nitrite solutions. According to the author, the results of these experiments constitute the first case showing that in an animal body nitrogen in the form of inorganic salts in foods can be converted into organic nitrogen. Haworth, MacGillivray, and Peacock [362] have studied the reaction of formaldehyde with proteins at pH 10 and have concluded that under these conditions formaldehyde converts some of the amide groups of

the protein molecule into *N*-hydroxymethyl derivatives. By using this method, it was shown that silk fibroin contains no amide groups. Ueno [373] has made a study of the solubility of sericin in the degumming of silk as a function of the pH of the solution and the concentration of soap used. It has been found [368] that water with high temporary hardness dissolved more sericin in reeling silk from cocoons and caused the fibers to stick together and form hard gum spots in the skeins. After neutralization with bicarbonate, the reeling was improved and was substantially as good as that obtained with soft rain or river waters. The breaking load of silk fibers [369] has been studied at 20°C and throughout a range of 31%–90%. The results indicate that the breaking load decreases from 17.7 to 13.0 g., with an increase in the extension to break of 26.6% and 41.1% as the humidity is increased. Ikoma [363] has reported on the composition of tannin-chrome compounds used for the treatment of silk to improve sun-resistance.

4. Regenerated and Synthetic Protein Fibers.—There has been active research interest in the field of synthetic protein fibers, both with respect to fibers made from natural non-fibrous proteins, such as casein, corn products, and peanut protein, and to fibers made from components which are synthesized directly from non-protein base materials. Lundgren [402] has prepared a detailed review of synthetic fibers made from proteins. The literature in this field has also been reviewed by Fröhlich [392] and Salquain [404]. Wormell [408–410] has reviewed the present knowledge about fibers made from dispersed proteins, as well as fibers made from regenerated wool. Regenerated wool fibers can be made by dispersing the wool keratin in a sodium sulfide solution, followed by an acid treatment to precipitate a curd of keratin. The purified curd is dissolved in ammonia and extruded through a spinneret into a neutral bath. Keratin can also be dispersed in a sodium sulfide solution to which casein is added to form a spinning solution for dye extrusion into a salty bath. Wormell has discussed the properties of yarn spun from this kind of regenerated wool fiber.

The properties of vegetable protein fibers have been considered by Karrh [398], Koch [400], and Sisley [405]. The preparation and properties of "Ardil" fiber made from peanut protein have been summarized by Gapp [393] and Kapur [397]. In order to provide softness to this finished fiber, plasticizers are added to the spinning solution; certain salts which act as strengthening agents are added to enhance the elasticity of the fiber. "Ardil" has a tensile strength of 15,000 lbs. per sq. in., and a breaking extension of 80%; resistance to abrasion is relatively poor. Arthur and Many [378] have described the construction and operation of a pilot plant for spinning "Ardil" and have given typical operating data for such a plant. Experiments with casein fibers to improve the water- and boiling-resistance have been described by Brintzinger and Wolff [383]. Hougen and Hipp [396] have studied the flow properties of casein as a function of pressure in the range 500–3000 lbs. per sq. in., in the temperature range from 50°–140°C, and with differences in water content from 8%–28%. They found that there was no significant difference between the flow properties of rennet caseins and high-grade acid caseins.

Efforts to stabilize synthetic fibers produced from natural proteins have centered on formaldehyde reactions with the fibers. Croston [387] has found that zein fibers may be stabilized by curing in the presence of an aldehyde and a strong acid. At 100°C, the cure is very rapid and produces irreversible formaldehyde cross-links. This type of stabilization reduces the shrinkage and provides improved strength of the fiber after boiling in acid dye baths. Croston and Evans [388] have described methods for stabilizing filaments spun from formaldehyde-treated alkaline dispersions of zein by baking at 160°C. Peterson and McDowell [403] have stated, however, that casein fibers hardened with formaldehyde solutions need further processing to withstand boiling acid dye baths. Their studies on desamination of casein fiber at several steps in the spinning process indicate that desamination increases the fibers' resistance to dye baths. The desaminated fiber is given further stability by treatment with acid-formaldehyde solutions, followed by baking at 110°C. It is reported that dye uptake of both acidic and basic dyes is reduced by these combined treatments. Tetlow [406] has reported on the reaction of groundnut protein with formaldehyde, and Traill [407] has considered the reactions of formaldehyde with fibers synthesized from natural proteins. He has discussed the possible reactions of formaldehyde with various functional groups of proteins, and has concluded that amino and amide groups are concerned in the reactions of formaldehyde with proteins and that guanidyl, hydroxyl, and sulphydryl groups may also be in-

volved. D'yachenko [389] has found that the adsorption of water from saturated vapor by casein and the integral heat of adsorption are not affected by treating casein with formaldehyde vapor, at least to the point where 25 mg. formaldehyde per g. of casein is taken up.

The synthesis of polypeptides has received considerable attention and forms an important part of the research on synthetic protein materials. Elliott and Ambrose [390] have studied the polarized infrared spectra of several of the new polypeptides (principally *D*-*L*-phenylalanine *L*-glutamic methyl ester copolymer) prepared by Hanby, Waley, and Watson. When such a peptide is prepared as a film cast from *meta*-cresol, the film shows an α-configuration which can be highly oriented. When films are cast from formic acid, a β-configuration with low orientation is produced. X-ray diffraction and infrared spectra show that there is a great similarity between such polypeptides and the naturally-occurring fibrous proteins. Elliott and Ambrose have found that the presence of a 5.2 Å. meridian spacing in the fibrous α-proteins is not necessarily incompatible with the α-fold suggested by Ambrose and Hanby. They have also pointed out that Astbury's view that an α to β transition must be accomplished by a 100% chain extension is not shared by other workers in this field, who have found that their experiments indicate that the β-type of pattern is produced at much less than 100% extension. The model proposed by Mizushima and coworkers for the α-keratin configuration, which consists of a mixture of folded and extended forms, does not appear to agree with the polarized infrared data reported by Elliott and Ambrose for studies of α-configuration films. In further studies on the infrared absorption characteristics of the C=O peptide link, Elliott, Ambrose, and Robinson [391] have reported that this band shows an increase in frequency when insulin is denatured. This is interpreted to be due to the change from the folded α-chain configuration in natured insulin to an extended β-chain configuration in denatured insulin. The films of insulin showing α-chain configuration were cast from a solution of *meta*-cresol, and those showing β-configuration were cast from formic acid. These authors have also reported that the transformation from the α- to the β-chain configuration occurs without marked changes in the packing of the side-chains, but merely by the rotation of the peptide group about bonds attached to the asymmetric carbon atom in the chain. Bamford, Hanby, and Happey [380] have treated oriented fibers of the copolymer of *DL*-β-phenylalanine and *DL*-leucine with carboxylic acids other than formic, and have found that there is little or no effect in the x-ray patterns of these fibers. After treatment by a number of carboxylic acids, such as acetic, propionic, *n*- and *iso*-butyric, and *n*- and *iso*-caproic acids, the fibers of this copolymer become completely insoluble in benzene at room temperature. This suggested that, since the insolubility was not connected with salt formation, some transformation to the β-form had taken place. By studying the infrared-absorption characteristics of films of this acid-treated copolymer in the neighborhood of 1,650 $cm.^{-1}$, evidence was found for the presence of the β-form, both in the stretching frequencies of the CO vibrations and in the NH deformation frequencies, as previously reported by Ambrose and Elliott. From these infrared studies, it is estimated that after treatment with acetic acid, about one-third of the copolymer exists in the β-form.

Studies of the reactions of *N*-carboxy anhydrides of α-amino acids have received active attention. The reaction of these anhydrides with simple organic bases has been shown to provide a mechanism for their polymerization and is a new method for synthesizing polypeptides by the addition of single amino-acid units. Chantrenne [384] has prepared *N*-carbobenzyloxyglycyl phosphate and has found that this reacts rapidly with amino acids with the formation of corresponding carbobenzyloxyglycyl-peptides; free peptides can be prepared from these compounds by catalytic hydrogenation. With this method, the following polypeptides have been produced: glycylglycine, glycyltryptophan, and glycylglycyltryptophan. Bailey [379] has described a new peptide synthesis in which *N*-carboxy anhydrides can be used to synthesize a wide variety of polypeptides through a reaction which leads to carbamate derivatives as intermediate products. For example, *N*:*N'*-dicarboxy di-anhydride of *L*-cystine in reaction with glycine ethyl ester produces the carbamate derivative of *L*-cystinyldiglycine ethyl ester, which decomposes on heating to give a free tripeptide ester. This ester in reaction with *N*-carboxy anhydride of *D*-*L*-alanine in the presence of one equivalent of triethylamine yields the ethyl ester of a new pentapeptide, di-*DL*-alanyl-*L*-cystinyldiglycine. With this method, Bailey has synthesized eleven peptides which are derivatives of alanine, gly-

cine, cystine, and tyrosine. Katchalski and Spitnik [399], influenced by the reported work on the polymerization of *N*-carboxy anhydrides of α-amino acids, were led to a study of the possibilities of synthesizing poly-arginine. They have reported the successful preparation of poly-arginine by the guanidization of poly-ornithine. The latter was synthesized by using the same reaction as that for the synthesis of its homologue, poly-lysine, previously reported by Katchalski and coworkers. Levy [401] has reported on a new method for producing anhydrides in *N*-carboxyphenylalanine by refluxing *DL*-β-phenylalanine with a solution of phosgene in toluene. Breitenbach and Richter [381] have reported on the preparation of peptides from the acid anhydride of *N*-phenylalanine and from the acid anhydride of *N*-methylglycine. Hofmann, Magee, and Lindenmann [395] have prepared a number of representatives of a new class of amino-acid derivatives which they have designated as α-amino acid carbobenzoxyhydrazides and which represent valuable intermediates for the synthesis of complex peptides. These compounds, through their free amino group, may be readily combined with other peptide structures to form peptide carbobenzoxyhydrazides. Hanes, Hird, and Isherwood [394] have described an enzymic reaction in which peptides are formed by the interaction of free amino acids and the tripeptide glutathione. Bresler and Selezneva [382] have reported that they have synthesized polypeptides by the condensation of amides of hydroxy acids. Cook and Levy [385, 386] have synthesized glycyl peptides using 2-thio-5-thiazolidone as a reagent for the addition of a glycyl residue to the ester of an α-amino acid; coupling is reported to take place in the presence of a tertiary base, and the product is released by acidification. These authors have also reported on the synthesis of alanyl peptides using 2-thio-4-methyl-5-thiazolidone. Alfrey, Morawetz, Fitzgerald, and Fuoss [377] have reported that they have synthesized a material which is closely analogous to proteins in its electrical behavior in solution. The material is a copolymer of 2-vinyl-pyridine and methacrylic acid, which is reported to contain, on the average, three (weakly basic) pyridine groups and two (weakly acidic) carboxyl groups on every ten atoms of the chain.

D. Structure and Properties of Synthetic Fibers

The development, structure, and properties of several synthetic fibers have been compared [418, 427, 437–439, 451, 459, 462]. In the study of synthetic-fiber structure, x-ray [414, 461] and electron diffraction techniques [421] have been used to determine crystalline size and orientation. A study of the dielectric properties of Terylene has been reported by Reddish [452]. Kobayashi [436] has followed the thermal degradation of some polyamides by the use of viscometric methods. Müller [449, 450] has reported the effect of temperature and mechanical conditioning on the stress-strain relationships of various synthetic fibers and films. The effect of hydrochloric acid on these relationships has also been studied by Lee and Umakawa [444].

Considerable attention has been devoted to the part that interchain hydrogen-bonding plays in fiber structure and physical behavior [411, 419, 455]. Beauvalet and Champetier [416, 417] have reported the preparation of polyamide fibers in which the chains contain hydroxyl groups to provide additional opportunity for strengthening through hydrogen-bond formation.

Reports on the preparation and properties of nylon and its foreign counterparts Perlon (German) and Amilan (Japanese) have been published. Perlon L [460] and Amilan [424] are made from phenol by chemical synthesis. The molecular weights of polyamides have been evaluated by end-group titration [415] and by making use of viscometric data [447]. Their mechanical properties have been extensively measured [412, 413, 445, 448]. The internal structure of polyamides has been studied with x-rays and the polarizing microscope by Gabler [423], and by Kordes and coworkers [440]. In a study by Kauffman and George [429] the change of x-ray structure with application of load was measured in an attempt to explain the unusual mechanical properties of these fibers.

Considerable interest has been aroused by the recent production of polyvinyl alcohol fibers in commercial quantities. These fibers have been studied extensively in recent years by the Japanese [430–434, 441–443], who have succeeded in producing the fiber by a wet-spinning process and stabilizing the structure by cross-linking with formaldehyde [463]. Mechanical tests on the fabrics made from the fiber are very encouraging [453]. MacGillavry [446] has reported an x-ray study of these polyvinyl alcohol fibers.

References to the alginate fibers (produced from seaweed) continue to appear in the literature [420, 457, 458]. Interest in these fibers lies in their ready

solubility in alkaline solutions which permits them to be used in speciality fabrics. Polyurethane fibers (Perlon U) have received attention by a number of German workers who investigated their crystal structure and orientation as well as their dye-absorption characteristics [422, 428, 464]. The production and properties of Orlon, the DuPont polyacrylonitrile fiber, were described by Houtz [426].

A number of new polymer combinations were reported to form fibers with properties worth further consideration. The Japanese have prepared addition polymers from polymethylene diisothiocyanates and polymethylenediamines in the class of polythioureas with tensile strengths as great as 1.5–2 g. per den. [425]. Somers has described the preparation of cyanoethyl ethers of cellulose by the reaction of viscose with acrylonitrile to produce a protein-like fiber [456].

Unfortunately, the improvements in glass fibers made in this country in recent years have not been adequately described in the technical literature. Only a limited discussion of glass-fiber technology appears in the German literature [435, 454]. Some real improvement in the problem of brittleness has been made but the exact methods and basis for this work remain an industrial secret.

E. High-Polymer Studies

1. General Papers and Theory.—A number of important publications, principally textbooks dealing with high polymeric materials, have appeared in the literature during the period covered by this report. These include publications by Alexander and Johnson [465], Houwink [470], Kruyt and Jackson [473], Mark and Tobolsky [475], Post [478], and Reiner [479]. Among papers dealing with the general theory useful in high-polymer research studies are a number concerned with the time-dependence of irreversible processes, such as the laws of viscous flow. Cox [467] has extended the method of Gibbs to the treatment of irreversible processes. Bridgman [466] has suggested that the concept of entropy can be generalized to apply to intrinsically irreversible processes in which the final state is not attainable from the initial state by an alternative reversible process. He has considered a plastic solid with the stress-strain hysteresis cycle as an example, and he has derived an expression for the total irreversible entropy increase arising from an irreversible change in a solid, where the degree of internal disorder may be specified by a single parameter, in addition to the pressure and temperature of the system. Mack [474] has developed a theory of anomalous viscosity and elasticity observed with plastic systems; the theory is based on the concept of an order-disorder arrangement of the molecules and the change in this arrangement under stress. James and Guth [471], in a simple presentation of the network theory of rubber, have discussed the form of an ideal stress-strain curve, making use of approximations implicit in the Gaussian network model. Katz [472] has made a thermodynamic analysis of stress in one-component, one-phase elastic systems and has suggested that the stress be evaluated in terms of entropy and enthalpy contributions. In this study he has indicated that the basic hypotheses of the kinetic theory of rubber elasticity lead to conclusions which are thermodynamically impossible.

Hadwiger [468] has considered the statistical problem of the most probable form of a chain molecule. Zimm and Stockmayer [480] have developed formulas for the mean square radii of various branched and ringed polymer molecules, using the conventional assumptions regarding the statistics of chain configuration. Their results have shown that the number of branches or of rings can be determined from light-scattering measurements. Porod [477] has proposed that the properties of threadlike molecules be evaluated in terms of a "persistence length," $a = l/(1 - K)$, where l is the length of the single link in the molecular chain, and K $(= \cos \alpha)$ is the mean value of the cosine of the valence angle between successive chain atoms. The mean separation of chain ends is shown to depend simply on the parameter α. Montroll [476] has used a mathematical method based on the theory of Markoff chains to eliminate short-range excluded volume effects in polymer chains formed on space lattices.

Herdan [469] has interpreted the differences between molecular-weight averages of polymers obtained by different methods in terms of inequalities, and has interpreted the relationship between these inequalities and the degree of polydispersity. This method permits the identification of three empirical relations between such averages and three fundamental algebraic inequalities. He has shown how to calculate the standard deviation and the higher characteristic parameters of the distribution of molecular weights from a knowledge of averages of different order.

2. Physical Properties.—Treloar [536] has recently surveyed the field of rubber elasticity and has presented both the principal experimental facts and the theoretical consideration underlying this subject. Leaderman [508] has summarized some general aspects of the mechanical properties of high polymers and has described the elastic and inelastic behavior of systems containing long-chain molecules. Making use of Boltzmann's principle of linear superposition, Sips [528] has described a general relation between force and deformation for a linear sample of a visco-elastic substance. With this relationship, he has shown how the usual properties, particularly creep and recovery, may be predicted. Rivlin [524] has presented further results in the development of a theory for torsion, shear, and flexure relating to large elastic deformations of isotropic materials. Rivlin [525] has also given a critical discussion of the usual vibration equation which is employed to evaluate the dynamic elastic modulus and viscosity coefficient in dynamical experiments on rubber. In connection with the theory of plasticity, White and Drucker [540] have emphasized the importance of "effective stress" and "effective strain." Mathematical methods of stress analysis have been presented by Read [523] for linear, compressible, visco-elastic materials such as metals at high temperatures or high polymers with small strains. Huggins [504] has considered the flow and elastic properties for crystalline solids, simple liquids, glasses, and high polymers in a qualitative review which emphasizes the comparative properties of these materials. Jenckel [505] has reviewed the plastic-elastic behavior of high polymeric materials and has shown how this behavior may relate to their chemical structure. The importance of molecular forces in the mechanism of elastic deformation [491, 539] has also been discussed.

Studies of the dynamic modulus and energy loss per cycle observed in the dynamic loading of high polymeric materials have received considerable attention during the past year. Treatments of linear visco-elastic systems have been made by Cini [488], Thorne [534], Roscoe [526], Gross [500], Skudrzyk [529], and Lethersich and Pelzer [509]. Pisarenko [522] has applied an approximate method of non-linear mechanics to the problem of finding the resonance curve of an oscillating elastic system, considering energy dissipation. Measurements of the dynamic properties of rubber and rubber-like materials have been reported by Fletcher and Gent [495], Hillier [501], Kuhl and Meyer [507], Mullins [513], and Nolle [519]. Parfitt [521] has reported on studies of energy dissipation in high polymers in the glassy state for the frequency range 5–60 kc./sec. In this frequency range, perspex, both plasticized and unplasticized, showed little change in the fractional energy loss per cycle and in Young's modulus. The losses in perspex were reported to be about six times those obtained with polystyrene. Nielsen, Buchdahl, and Levreault [518] have studied the dynamic mechanical and A.C. electrical properties of plasticized polyvinyl chloride; and Merz, Nielsen, and Buchdahl [512, 517] have reported on the mechanical properties of cast polystyrene films. Warner [538] has described the use of electrical measurements to predict the mechanical properties of plasticized polyvinyl resins. Marvin, Fitzgerald, and Ferry [511] have made measurements of the dynamic viscosity and rigidity of polyisobutylene at frequencies from 20 to 600 c./sec. The rigidity increases and the viscosity decreases with increasing frequency, while both decrease with increasing temperature and appear to be almost independent of the molecular weight of the material.

Findley [494] has given consideration to the relationship between creep and the damping properties of polystyrene. Ferry, Sawyer, Browning, and Groth [493] have measured the dynamic rigidities and viscosities of concentrated solutions of polyvinyl acetate in 1,2,3-trichloropropane. Képès [506] has described an apparatus for studying the elastic modulus and the internal friction of solids and has reported results on polyvinyl chloride obtained with this equipment.

The creep characteristics of plasticized polyvinyl chloride have been studied [481, 520], as well as the creep-time relations for polystyrene subjected to bending, tension, and torsion [510]. Conant, Hall, and Lyons [489] have reported on an empirical equation which has been found to represent the time-temperature dependence of visco-elastic behavior of Hevea, GR-S, Neoprene GN, Butaprene, and butyl rubber. The authors point out that a temperature dependence of the activation energy is indicated, when this empirical equation is compared with that derived by Tobolsky and Eyring using the absolute reaction-rate theory. Buchdahl and Nielsen [486] have reviewed the evidence for and against the interpretation of the "second-order" transition in terms of a thermodynamic transition of the second order. They

have concluded that, of the various molecular processes responsible for thermodynamic transition, those relating to the general mechanism of "hindered" rotation are most likely origins of the second-order effects. Fox and Flory [496] have related the second-order transition temperature, viscosity-temperature coefficient, and specific volume through an empirical expression to the molecular weight and temperature of polystyrene. Evans, Mighton, and Flory [492] have studied the dependence of melting temperatures of polyesters and polyamides as a function of composition and molecular weight. Brenschede [485] has studied the transformations in synthetic linear high polymers formed by rapid cooling of a melt; the transition from a glassy state to a crystalline state on aging was determined by measurements of the density, double refraction, and x-ray diffraction patterns of the material. He has concluded that the development of such a crystalline form from a glassy state does not result from the presence of a seed crystal in an amorphous region but appears to develop groupwise by a spontaneous gathering of one ordered region with another neighboring one.

Gehman, Jones, Wilkinson, and Woodford [499] have studied the progressive stiffening of natural and synthetic rubber vulcanizates at low temperatures and have related changes in the measured torsional stiffness to the degree of crystallization determined dilatometrically. Refractive index-temperature data have been reported for a series of rubber-like polymers down to a temperature of −120°C [541]. The second-order transition temperature was shown to be located by a change in the refractive index-temperature slope. Würstlin [542] has determined the second-order transition temperature and the brittle point by measuring the dielectric constant and the dielectric loss factor as a function of temperature for a number of linear polymers.

Boyer [484] has reported on the detection of random noise in dielectric materials, particularly thin films of polar plastic materials, which have been subjected to direct-current potentials of 10 to 300 v. per mil. The fluctuations which he has reported are about 1,000-fold above the noise level of the circuit and are about 1% of the steady D.C. current. The noise is most pronounced in materials such as cellophane, nylon, and copolymers of vinylidene chloride with acrylonitrile, and is presumably caused by the motion of the polar groups in the structure. The heat conductivity of both natural rubber and GR-S has been studied in the range from +50°C to −170°C, and from 0% to 100% elongation. Above and below the second-order transition region, the heat conductivity decreases approximately linearly with temperature as predicted by classical theory [490]. However, the variation through the second-order transition does not agree with classical theory, but may be explained qualitatively on the basis of a diffuse lambda type transition. Saunders [527] has found, for rubber vulcanized by a peroxide reaction, that the stress and birefringence are linear and are reversible up to strains of about 100%. He has reported the stress-optical coefficient to be 2.05×10^{-10} cm.^{2}/dyne. Fox, Flory, and Marshall [497] have measured the elongation at which crystallization starts in vulcanized rubber by changes in density, and have found that the critical elongation for incipient crystallization depends on the temperature, and the crystallization sets in at an elongation well below that at which the stress-strain curve assumes a steep slope. Villars [537] has reported the development of a high-speed stress-strain machine which is capable of recording the stress-strain curve of elastomers at elongation rates up to 270% per μsec.; he has reported some interesting studies using this equipment on such materials as gum and tread stocks of hevea, and such synthetic elastomers as GR-S, Hycar OR, Butyl, Perbunan, and Neoprene GN.

Boonstra [483] has described some interesting experiments on the stress-strain properties of natural rubber under biaxial strain. Tube-shaped samples were stretched in a direction parallel to the tube axis and at the same time inflated to cause a tangential extension. In using this technique, 600% axial and 300% tangential elongation could be attained simultaneously. Hopkins, Baker, and Howard [502] have also applied the use of biaxial stress to polyethylene and have described an apparatus for the complex stressing of both sheets and tubes. A study of the fundamental and practical mechanical properties of various molecular-weight polyethylene resins has been reported by Carey, Schulz, and Dienes [487]. Isothermal-compression measurements on polymethyl methacrylate, polyethylene, cellulose acetate-butyrate, and ethyl cellulose have been reported by Spencer and Gilmore [531]. Spencer [530] has also considered the relation of the Newtonian viscosity and the modulus of elasticity to the flow behavior of linear amorphous polymers in connection with his work on

the behavior of polymers in shear. Tohara [535] has developed a new measure of stress-relaxation, taking into account the transient concentration of relaxing stress. van Amerongen [482] has made a study of the effect of structure of rubber-like materials upon the permeability, diffusivity, and solubility, using a series of gases. Gee, Stern, and Treloar [498] have measured volume changes in the stretching of vulcanized natural rubber and have found that the small expansions observed are in agreement with those calculated from the known compressibility of the rubber and the hydrostatic component of the stretching force. The velocity of propagation of ultrasonic waves in macromolecules has been reported by Natta and Baccaredda [514–516]. With polythene, polyisobutylene, polyalpha-butene, polystyrene, and polyvinyl isobutyl ether it was found that the ratio (R) of the ultrasonic velocity to the density of various polymers is related to the form of the molecules. In compounds with a straight paraffin chain, R increases with increasing molecular weight; in cross-linked compounds with ethyl and methyl groups, R is practically a constant for all compounds up to a molecular weight of 15,000. A similar series of studies by these authors was conducted with 66 nylon, natural rubber, butyl rubber, and a number of other copolymers of rubber-like materials. The experimentally measured velocity of propagation of ultrasonic waves was compared with the velocities calculated both on the basis of the bond velocities of Lagemann and Corry and on the basis of the radicals that make up the molecule. Better agreement between the calculated and experimental values of ultrasonic velocity is found if radical velocities rather than bond velocities are considered.

Other papers relating to the physical properties of high polymers are concerned with crazing [530], with the development of surface cracks as affecting strength properties [533], and with frictional forces between polymer and channel walls which produce granulation during the extrusion and injection molding of high polymers [532].

3. Polymer Structure.—A summary of the "Proceedings of the Conference of the X-Ray Analysis Group on Macromolecular Structures" [543] includes a review of papers given on the application of small-angle x-ray scattering to colloid-structural problems, on studies of the structure of synthetic polypeptides, and on the x-ray investigations of reaction mechanisms in the substitution of cellulose. This summary contains 37 references to the published literature.

Lifshits and Rozentsveĭg [552] have considered the theory of scattering of x-rays by elastically strained polycrystalline materials and have presented a general equation for the displacement of the diffracted line in terms of the strain imposed on the crystallites of the material. Hermans and Weidinger [549] have made a quantitative study of the x-ray diffraction produced by amorphous polymers. Arlman and Goppel [544, 545, 548] have studied the degree of crystallinity in natural rubber and have correlated their x-ray results with density measurements. The maximum value of 30% crystallinity deduced by Goppel has been corrected upward to approximately 37%. These results are compared with the unit cell proposed by Bunn and have been discussed in connection with the work of Flory, Gee, and Wildschut. Clark, Mueller, and Stott [546] have studied the structure and physical properties of massive nylon, making use of x-ray diffraction, photomicrography, hardness and density measurements, heat treatment and recrystallization in various liquid media, and tensile measurements. A study of polyethylene by electron diffraction [556] has confirmed the existence of the identical structure of the hydrocarbon chain contained in macromolecules of polyethylene and in saturated hydrocarbons. Parameters for the orthorhombic crystal structure ($a = 7.38$ Å., $b = 4.92$ Å., and $c = 2.51$ Å.) are the same as those for polyethylene obtained by x-ray diffraction and for paraffin chains by electron diffraction. Infrared studies [547] of polyvinyl alcohol have appeared to indicate that in the fundamental O—H bands, there may be evidence for different assigned positions of the hydrogen atoms from those concluded by earlier x-ray studies. The presence of dichroic bands caused by bound water is also found in the region of the O—H band. This complicates interpretation and may explain the discrepancy between the results of this work and that of Elliott, Ambrose, and Temple. Signer [555] has discussed methods of interpreting gel-permeability and streaming-birefringence determinations and electron micrographs in terms of the molecular structure of micelles and fibers. X-ray diffraction studies of dissolved chain molecules [551] indicate that the scattered radiation is divided into four regions with increasing scattering angle; the first is related to the whole coiled molecule, the second to smaller coiled portions,

the third to straight portions of the chain, and the fourth to single atoms or monomer units which cause scattering at the largest angles. The length of a statistical chain element may be estimated from the second and third regions, and that of a monomer unit from the fourth region. The results of such studies have been reported in connection with polyvinyl bromide and cellulose nitrate. Karpov and Kargin [550] have used the line width of a beam diffracted by a crystal lattice to determine the particle size of crystallites. Winogradoff [557] has observed that when samples of polystyrene, polythene, and paraffin wax have been irradiated with x-rays, they display a bluish-white luminescence in an x-ray beam, and polythene and polystyrene become discolored after prolonged radiation. Prolonged exposure to x-rays produced strong absorption of ultraviolet radiation in polystyrene. Mochel and Hall [553] have studied the infrared absorption spectra of amorphous and crystalline Neoprenes, Type CG and Type GN, in the wave-length range 2–23 μ. The spectra appeared to be unchanged by variations in molecular weight or sol-gel relationships. The authors have reported, however, that pronounced spectral changes accompany crystallization of Neoprene either in the unstretched state or following stretching. Perceptible spectral changes were found several days after stretching of Neoprene GN, which appears to crystallize slowly.

Selwood, Parodi, and Pace [554] have found that measurements of the magnetic anisotropy of substantially amorphous polymers may be applied to study changes in molecular orientation. These authors have measured the magnetic anisotropy of crystals of dimethyl terephthalate, and by assuming that the monomer is the principal repeating unit in polyethylene terephthalate, they have been able to interpret the data of their magnetic susceptibility and anisotropy measurements. Measurements made at 25°C on polyethylene terephthalate fibers drawn at 91°C indicate that there is a 25-fold increase in the magnetic anisotropy as the draw ratio is increased 5-fold. Magnetic susceptibility and anisotropy measurements were also made on polyethylene terephthalate which had undergone various conditions of heat treatment.

4. High-Polymer Solutions.—Staverman [578] has derived mathematical formulas for the free energy of high-polymer solutions, extending Huggins' theory to include application to molecules containing rings and cross-links. Flory and Krigbaum [565] have derived thermodynamic relationships which are applicable to dilute solutions of heterogeneous high polymers. Münster [572] has developed a theory of solubility of high-polymer fiber molecules, based on the thermodynamics of polydisperse solutions.

A general theory of the Rayleigh light-scattering due to composition fluctuations in multi-component systems has been developed by Kirkwood and Goldberg [567] and applied to interpret the turbidity measurements of Ewart, Roe, Debye, and McCartney on polystyrene in benzene-methanol mixtures. Stockmayer [579] has independently derived a theoretical treatment of the same subject. Stockmayer and Stanley [580] have reported on light-scattering studies of the interaction between polymethyl-methacrylate and polystyrene in butanone at 25°C. Doty and Steiner [562] and Boyer and Spencer [560] have studied critical-concentration effects in dilute high-polymer solutions. The critical concentration may be related to the effective diameter of the randomly coiled molecules in solution. The effective diameter of solvated polymer molecules may be estimated by light-scattering experiments [574]. Debye and Bueche [561] and Outer, Carr, and Zimm [576] have used light-scattering to study solutions of polystyrene. Adelstein and Winkler [558] have reported on studies of the diffusion of polystyrene in different solvents. The activation energies for diffusion were found to be of the order of 3 kcals. per mole, and the results, in general, support Eyring's theory of segment flow for polymer diffusion. Dunning and Patterson [563] have studied the flow properties of dispersions of latex rubber in Aroclor 4465; the form of the compression-time curves and the order of magnitude of the strain-relaxation time agreed with the theory of Fröhlich and Sack.

Montgomery and Winkler [569–571] have studied the polymerization and depolymerization of polystyrene, the synthesis and degradation of polystyrene possessing the Kuhn-Schulz chain-length distribution, and the scission of polystyrene by a variety of agents. The depolymerization of polystyrene and carboxymethylcellulose has been studied by Prudhomme and Grabar [577] using viscometric measurements. It was found that polymers of the same chemical composition but of widely differing molecular weight were broken down by the ultrasonic waves to the same molecular weight or chain length. These authors have stated that chemical effects

produced by ultrasonic radiation do not enter into this degradation. Merz and Raetz [568] have described a large-scale fractionation of a low-conversion polystyrene polymer by reducing the temperature stepwise in a butanone-methanol solution. The results of measurements of intrinsic viscosity for all 39 fractions and the light-scattering determinations of the molecular weight of 8 fractions have been reported. Batzer [559] has obtained polyesters having a molecular weight of 1,000,000 by a new method of polycondensation. Ostroski and Stambaugh [575] have described the application of ultrasonic radiation to emulsion polymerization of butadiene. They found that the intensity of the sound source must exceed a critical value before any appreciable accelerating effect is observed. Above this critical intensity, the authors have stated that the time to reach a given yield of polymer is about inversely proportional to the power used.

Nitsche and Toeldte [573, 581] have suggested that the solubility characteristics of high-molecular-weight polymers may be used to characterize these materials. Dielectric measurements have been conducted by Funt and Mason [566] on solutions of polyvinyl acetate in toluene at three fixed frequencies. The molecular weight and the solution concentration were systematically varied, and it was found that the dielectric behavior of polyvinyl acetate in solution was in better agreement with the Debye theory than that generally found for solid polymers. Wall, Stent, and Ondrejcin [582] have developed a simple theory for the electrical transference properties of polymeric acids. Experiments with polyacrylic acid and on the hydrolyzed copolymer of maleic anhydride with styrene have indicated qualitative agreement with the proposed theory. Edelson and Fuoss [564], as a result of studies of viscosities of polymethyl acrylate in dioxane, tetrabutylammonium polyacrylate in methanol, and sodium polyacrylate in water, have concluded that the unusually high viscosity of sodium polyacrylate solutions in water is due to cross-linking of polyacrylate ions through hydrogen bonds with water molecules. This is an important and probably general result by which it may be stated that polymers containing groups which can bond to hydrogen will give much higher viscosities in water than in other protonic solvents because water is bifunctional.

F. Fiber, Yarn, and Fabric Inter-Relationships

The major research effort in this field has been study of how fiber or filament properties and fiber organization affect the properties of the resulting yarns. The question of what fiber properties are important in the determination of yarn properties cannot readily be divorced from the complications of yarn structure. Yet this separation is still being attempted in the correlation studies of Webb and Richardson [656, 657] and is summarized by de Meulemeester [624] with some measure of success because a limited number of variables in yarn structure have been used. This work and others [612, 616, 617] have emphasized the importance of the fiber-length uniformity ratio [628], and indications are that this property in staple fibers may have greater significance than hitherto suspected. Beste and Hoffman [589] have continued their study of factors contributing to the property of resilience.

Some excellent work has been done on the problems of yarn geometry and structure. In the simpler cases of continuous-filament yarns, the factors of twist, strength, elasticity, stiffness, and fiber denier in determining the stress-strain characteristics of yarns have received attention [593, 598, 618, 631, 632, 643, 645]. The problem with staple fibers is more complicated, but a notable beginning has been made by Gregory [609–611], who has investigated the relation between bulk and twist and the strength of cotton-yarn elements. Platt has studied not only the factors related to strength but those affecting elastic performance as well [633]. The influence of twist on yarn during tension has led to the concept of a critical yarn diameter [585, 586] which is that of the yarn element at maximum density; this concept may be used to evaluate yarn strength for a given material. A preliminary investigation of the twist-bulk relationship in jute has also been reported by Chakrabarti [595].

The effects of moisture [619] and rate of extension [623, 625] on yarn properties have been further studied.

Numerous papers have been published on the causes and effects of yarn irregularities. The causes are manifold: some are due to the nature of the fibrous material used [594], some to the inadequacies of the carding and spinning equipment [597, 599, 603, 620, 648–650], others due to uneven strains in the filaments during twisting [583]. Some effects

of these irregularities in rayon fabrics [621], knitted wear [655], and hosiery [652] have been noted.

The problem of measuring yarn irregularities is a complicated subject. An unevenness test may evaluate the weight per unit length of a yarn satisfactorily, but the problem of statistical and practical interpretation remains. More studies have been made along this line, and a number of approaches to the problem have been reported by Graf and Henning [607] and by Reichenbach [638]. Stout has established the relationship between incidence of break and applied tension for a given count [646]. Once this has been determined, it is only necessary to subject a measured length of the yarn to a certain minimum tension and count the number of breaks to have a complete evaluation of the distribution of weak places in the yarns. This method has certain obvious advantages over the commonly used skein strength which Vreedenberg has shown to be reliable only with very regular yarns [654].

The Textralizing process for artificially crimping fibers [584, 634, 636] also modifies yarn and fabric properties [629], although the complete evaluation of this process has still to be made.

Very little has appeared on the relation between yarn and fabric properties. Some progress appears to have been made in the field of tensile strength [590], but much remains to be done. Morton [626] has outlined the relation between yarn structure and its suitability for the production of rigid webs, tapes, and ribbons.

The measurement of a number of important fabric properties has been reported. These include studies of air porosity [591, 639, 640], stress-strain characteristics [615], moisture-absorbency [592], fatigue [596], felting [613, 614], shrinkage [606, 642, 658], and abrasion-resistance [601, 604].

Properties of blends of fibers are receiving increasing attention. Some blends reported on during the year 1950 are wool-nylon mixtures [637, 651, 659], wool-rayon mixtures [600, 641, 653], rayon-cotton blends [608], and Dynel blends [647]. Obviously, these are not the only combinations possible, and it is expected that the literature in this field will reflect interest in other natural and synthetic fiber blends.

This section would not be complete without some mention of the extensive work done in the field of clothing properties. The main efforts have been on studies of heat retention and air permeabilities of fabrics, especially those required in military uniforms. As yet, most of the emphasis has been on requirements, but it is to be expected that soon these requirements will result in modified and improved fabric designs [587, 588, 602, 605, 622, 627, 630, 635].

The expression of textile designs in terms of an enumeration problem, begun by Shorter last year, has been completed [644].

G. Adsorption, Swelling, and Diffusion

1. General Papers and Theory.—A brief general review of all types of adsorption phenomena with many references has been made by Harris [685]. The Faraday Society has published a general discussion on heterogeneous catalysis [679]. Although no work specifically applied to fibrous materials appears, there are several interesting articles on the theoretical aspects of surface interactions and on new experimental techniques.

Hill [690] and Everett [676] have considered the general thermodynamic problems involved in adsorption and have concluded that methods similar to those used for solutions are most convenient. Everett has extended his ideas to the treatment of adsorbed monolayers [677] and to the analysis of experimental data [678]. Hansen [684] has also discussed the analysis of experimental data, in particular the calculation of heats of adsorption from adsorption isosteres.

Tompkins [716, 717] has outlined a general approach to adsorption on nonuniform surfaces for both localized and nonlocalized monolayers. Hüttig and Theimer [694] have shown the relationship between Hüttig's multilayer adsorption isotherm and the BET isotherm, and Fergusson and Barrer [680] have given a statistical derivation for Hüttig's isotherm. Hill [689] has considered nucleation of gases in capillaries and on small particles, while Pierce and Smith [704] have discussed the relationship between capillarity and adsorption-desorption hysteresis.

The specific thermodynamic problems occurring when the adsorbent is elastic were treated by Hill [691]. Treloar [718] has calculated the effects of various types of homogeneous strain on the adsorption and swelling of an isotropic amorphous rubber-like network. Flory [681, 682] has extended his treatment of the swelling of networks to more highly cross-linked systems. Barkas [663] has presented

his current views on the swelling of polymers, in particular, wood, under stress in a series of articles.

Since the theory of diffusion was not extensively considered in last year's review, several of the more interesting papers of 1949 are reviewed here, together with those which have appeared in 1950.

Babbitt [662] has examined the fundamental bases of the differential equation for diffusion. The definition of the diffusion coefficient was considered by Hartley and Crank [687] with particular reference to systems where a net mass flow across the observation section occurs. Crank and Henry [671] have developed the diffusion relationships for certain cases in which the diffusion coefficient varies with the concentration, and Crank [670] has discussed the influence of concentration-dependent diffusion on the rate of evaporation. Danckwerts has developed equations for a system in which there is simultaneous diffusion and chemical reaction [672] and has also given a general method of solution of a class of problems involving two phases separated by a moving plane boundary [673]. Laidler and associates [698, 699, 709] discussed the mechanism and kinetic laws for diffusion through a membrane and have applied their results to the diffusion of sugars through collodion membranes.

2. Interactions with Simple Liquids and Vapors.—King [697] has presented a critical survey of recent work on adsorption and swelling and on properties dependent on these factors, in particular the electrical properties of wool and nylon. Hoover and Mellon [692] have obtained good results in the application of a theory, based on considerations of dipole-dipole interaction, to the adsorption of water on a variety of polar polymers. Dole [674] has discussed the application of his multilayer adsorption equations to the interaction of several synthetic polymers with water. Dole and Faller [675] have also considered the adsorption of water by several polyesters and polyamides. The adsorption by the polyesters is small and probably on end-groups only; with polyamides it occurs throughout and is sensitive to the degree of disorder.

Abbott [660] observed an anomalous effect of greater axial than lateral swelling for nylon which aroused considerable comment on its possible causes and implications [705, 708, 711, 721]. Zettlemoyer, Chand, and Gamble [722] measured the sorption of krypton and nitrogen on polyethylene, nylon, and collagen and found good applicability of the BET theory. Hunt, Blaine, and Rowen [693] examined the nitrogen adsorption of cotton presoaked in caustic and found greatly increased surface areas which they interpret in terms of pore-size distribution. Moore, Scott, de Gruy, and Rollins [702] have observed the swelling of cotton in water microscopically and have reported that there are no differences with respect to variety or maturity. Wahba [719] has determined the dependence of the heat of wetting of viscose rayon and standard cellulose on the moisture regain, and Wahba and Nashed [720] have discussed the change of the heat of wetting with temperature. The effects of drying conditions on the subsequent adsorptive and swelling properties of cellulose have been considered by Lauer, Bezner, and Dobberstein [700]. Stamm and Tarkow [715] have studied the penetration of cellulose fibers by liquids and the formation of aerogels, and Stamm [714] has examined the mechanism of binding of water.

Studies of the surface area and water adsorption of starch have been reported by Hellman and Melvin [688]. Rao, Rao, and Vasudevamurthy [707] showed that the sorption-desorption hysteresis of sericin is made permanent by treatment with hardening agents such as HCHO. Guthrie [683] has observed sorption hysteresis effects for cotton yarn on cycling over a small range of relative humidity.

Swelling and diffusion of liquids into cellulose acetate sheets has been followed by Hartley [686], who reported faster penetration perpendicular to the preferred molecular axis than along it. Simril and Hershberger [712, 713] have also studied permeability of polymeric films as a function of composition, plasticizer, and moisture content for a number of gases. Burlet [667] has observed the rates of adsorption and desorption of water by nylon. Alexander and Meek [661] have studied the drying of textile fabrics by high-frequency heating.

3. Interaction with Solutions.—Interaction with solutions of interest in connection with dyeing are given in the separate section on dyeing.

Champetier and Néel [668] have investigated the interaction of cellulose with water-sodium hydroxide, water-pyridine, and other binary mixtures, and have interpreted their results in terms of the formation of molecular compounds. Kasbekar [696] has examined the swelling of cellulose in several neutral salt solutions and has reported that it varies with the size of the solute particles and other factors. Karnik and Devadatta [695] have studied the shrinkage of

cotton yarns resulting from application of various acid and salt solutions.

Bowes and Kenten [665] found an increase in swelling of collagen at high pH values using NaOH and $(CH_3)_4NOH$, and Bowes [664], studying bivalent bases, found considerably less swelling than with NaOH. Brintzinger and Wolff [666] have discussed the swelling of casein in salt solutions.

4. Experimental Methods.—Newns [703] has reviewed experimental methods of determining the water-vapor permeability of laminae, and Mason [701] has prepared a review of the techniques for measuring the specific surface of fibers. The use of glass springs for adsorption measurements has been considered by Chipalkatti and Giles [669]. Preston and Nimkar [706] have outlined the use of the centrifuge method for measuring swelling of fibers and have suggested the use of surface-active agents to reduce the amount of water held by capillary forces between fibers. Signer and Roth [710] have developed a wick method for measuring the rate of evaporation of water from bundles of various fibers and have compared several types of fibers in this way.

H. Dyeing

Vickerstaff, in his recent book *The Physical Chemistry of Dyeing* [809], has reviewed the entire field of dyeing from a physical-chemical point of view. As a summary of the present status of the attempt to bring the art of dyeing into a quantitative framework of molecular interactions by an author who has contributed considerably in the field, this book should be useful to anyone interested in research on dyeing.

1. Properties of Dyestuffs and Dye Baths.—Knight has discussed the recent trends in the search for new azo dyes including dyes for wool [758], dyes for acetate rayon and nylon [759], and direct dyes for cotton [760]. Seidenfaden [798] has also reviewed the newer dyestuffs. Shah, Tilak, and Venkataraman [799, 800], in connection with the structure of sulfur dyes and sulfurized vat dyes, have proposed a new structure for hydron blue and have found that Cibanone Yellow R, previously assumed homogeneous, splits into fractions on chromatographic analysis. Mitchell [774] has published a short note on the crystallization of Congo Red.

Kuhn [764] developed the quantum mechanical theory of the relationship between the constitution and color of dyes, using an adaptation of the free-electron gas model for metals; he has obtained good agreement with experiment for some dyes. Other dyes require further extension of the theory [765, 766]. Kuhn [763] has also summarized the entire field of the quantum mechanical theory of color.

The color and constitution of azo pigment dyes have been related by Glassman [753]. Schetty [795] has examined the connection between the constitution of *O*, *O'*-dioxyazo dyes and their suitability for the metachrome process. Race [788], in considering the suitability of dyes for the metachrome process, has correlated it with the rate of formation of the chromium complexes of the dye. Specklin [801] has reviewed the metal-complexing properties of azo dyes.

Susich [804] has outlined methods of identifying dyestuffs by x-ray powder diffraction. Rao, Shah, and Venkataraman [789] described various methods of separating mixtures of acid dyes, basic dyes, and direct dyes by chromatography on filter paper, cellulose acetate, nylon, and vinyon fabrics. Richardson [791] discussed the diffusion and adsorption of direct cotton dyes in acid form in anion-exchange media. Chmutov and Yushkevich [744] have developed a new micromethod for measuring diffusion coefficients.

A new method of determining the activity coefficients of surface-active materials in aqueous solutions from surface-tension considerations has been outlined by Scholberg [796]. Colichman [745] has shown that the critical concentration for micelle formation is identical with the concentration necessary to suppress polarographic maxima.

Michaelis [772] has described recent work on molecular aggregation, on reversible polymerization in solution, and on changes brought about by additional solutes in terms of changes in the absorption spectra. Kravets, Pes'kina, and Pribytkova [762] deduced from theoretical considerations of the "half-band width" of the absorption bands for many dyes that these bands are composed of several narrow elementary bands. Freeman and Stein [751, 752] studied the variations with pH of the absorption spectra of some dyes in alcohol-water solution. Martin and Standing [767, 768] have examined the effects of sodium chloride, pyridene, ethyl alcohol, polyethylene glycols, and polyethylene oxide condensates on the absorption spectra of certain azo dyes; their results have been interpreted in terms of molecular complexes and micelle formation. Carroll and Thomas [741] found distinct changes in the absorption spectrum of Orange I in the presence of

hydrous aluminum oxide. They have used these changes to measure the ability of competing ions to form complexes with the sol. Merrill and Spencer [771], in a study of the effects of salts on the absorption spectrum of pinacyanol chloride in water solution and in sodium silicate solution, have concluded that salts have further effects than merely altering the degree of aggregation. The absorption spectrum of benzoylaminoanthraquinone vat dyes has been discussed by Waters [810], who has measured specifically the influence of the oxidation-reduction process and of soaping. A study of the absorption spectra of a variety of dyes in the form of solid films has been made by Vartanyan [807].

Bromberg [738] determined the diffusion coefficient of several dyes in gelatin gels and measured the deviation from Fick's Law. Bromberg and Mal'tseva [739] have discussed the effect of adsorption on the rate of diffusion in gelatine, while Veidenbakh and Karpovich [808] have studied the changes in the absorption spectrum of dyes in gelatine. Nishi [781] considered the dependence of the velocity of dialysis on temperature and particle size.

2. Interaction of Dyes with Fibers.—Meggy [770], in the seventh John Mercer Lecture, reviewed recent developments in the theory of dyeing. Royer [793] has also reviewed the recent fundamental scientific progress in dyeing and has stressed some examples of how these advances may be put to practical use.

(*a*) Polyamide Fibers: McGrew and Schneider [769] confirmed the hypothesis that acid dyes are attached to nylon by salt linkage with the basic amino end-groups. Remington and Gladding [790] have successfully applied a modification of the theory of Gilbert and Rideal to the quantitative interpretation of the uptake of acid dyes by nylon and have shown that hydrolysis at low pH produces new end-groups and, hence, new dye-adsorption sites. Munden and Palmer [777], using a series of 66 nylon samples of varying orientation, tested differences in dyeing properties and found the rate of dyeing to be dependent on the degree of orientation. For Perlon, Anacker [728] has reported that the adsorption of dibasic acid dyes is less than that of monobasic dyes.

(*b*) Wool and Protein Fibers: Alexander [724] has reviewed the dyeing of wool. Alexander and Kitchener [727] have discussed theories of wool dyeing and emphasize the role of the electric double layer. Elöd and Fröhlich [748] have studied the influence of temperature on the uptake of acid and substantive dyestuffs by wool, silk, and Ardil. Alexander and Hudson [726] have stated that at low dye concentrations, diffusion across a liquid film to the fiber is the rate-controlling step, while at high concentrations diffusion through the fiber is rate-controlling. Alexander and Charman [725], as a result of considering the kinetics of dyeing with a dye having a long hydrocarbon chain, have suggested that the surface activity of the dye contributes to its fast initial rate and high affinity. Olerup and Lindberg [782] showed that the rate of dyeing is remarkably dependent on the physical nature of the material being dyed; it is slower for wool fabric than for loose fibers.

Benson and Larose [733] studied the effect of sodium sulfate on the uptake of Orange II over a range of pH's. The theory of chromium and aluminum mordants for wool has been discussed by Justin-Mueller [757]. The leveling properties of acid and Palatine-fast dyestuffs on wool have also been examined [730]. Bhat, Kothare, and Nadkarny [734] observed the effects of electrolytes and nonelectrolytes in wool dyeing. Fern [750] outlined the correlation between the affinities of individual dyes and their compatibilities and leveling properties in mixtures. Millson and Turl [773], in microscopic studies of the influence of the cuticle in wool dyeing, found that distortions, such as knots, tend to dye more rapidly than the undistorted fiber. Burton and Stoves [740] have reported that various shades of brown can be obtained on wool by dyeing with colorless 2:4:5-trihydroxytoluene and subsequent treatment with metal salts. Bird, Nanavati, and Stevens [735] have conducted studies on the distribution of a direct dye between wool and cotton in one-bath union dyeing.

(*c*) Cellulose Fibers: Preston and Su [786] have indicated that the degree of polarization of the fluorescence of dyed fibers can be correlated with their dichroism, and Preston and Tsien [787] have used this method to derive orientation factors for the fiber and compare them with values derived in other ways. Elöd and Frölich [749] have examined the effect of fiber diameter and "skin" on dye uptake, swelling, and other properties. The present state of the theory of the diffusion of direct dyes into cellulose has been reviewed by Crank [746].

Pinte, Rochas, and Essertel [785] considered the relationship between dyeing affinity and degree of stretch in viscose rayon. Müller [775] has studied

the affinity and leveling properties of indanthrene dyes on cotton and staple fiber. Köster [761] investigated the dyeing properties of substantive cotton dyestuffs. Eckert and Abend [747] have discussed the acid dyeing of rayon treated with a resin of PhOH, urea, HCHO, glycerine, and phthalic acid. Bräuer [736, 737] has presented data on the use of cation-active compounds as aftertreating agents for substantive dyes. Cegarra [742] has reported on studies of the effect of temperature on the uptake of several dyes; maximum uptake at 60°C was observed for Dark Green BN. Chave [743] has applied the Freundlich isotherms to the uptake of dyes of viscose rayon, and Rosset and Paris [792] have studied the affinity of direct dyes for partially and completely saponified cellulose acetate. The dyeing of terylene polyester fibers has been discussed by Waters [811].

(*d*) Vat Dyes: The Committee on the Dyeing Properties of Vat Dyes of the Society of Dyers and Colourists [729] reported on standardized tests for migration and strike leveling. Müller [776] has made a review of fundamental research and practice in vat dyeing. Speke [802] has examined the restraining effect of Dispersol VL on leuco vat dyes. Abozin [723] used electrometric analysis to study the interaction of wool and cotton with the sodium salts of leuco indigo during vat dyeing. Wengraf [812] has discussed the mechanism of the action of protective colloids in vat dyeing, and Speke [803] has recommended the use of dextrin to prevent the over-reduction of temperature-sensitive colors.

(*e*) Printing: Torinus [806] found that Canarin, a yellow coloring material, can be produced directly within a fiber by printing a mixture of ammonium thiocyanate and an alkali chlorate. Canarin can also be printed beside other dyestuff groups. Barth [732] has studied the chemistry of chrome mordant printing. Witschonke and Fordemwalt [813] have described two analytical methods for determining the vat-dye content of prints. Barrick [731] related chemical constitution and printing performance for vat dyes. Munshi and Turner [778] studied the transfer of dye from the thickener to the fiber substance during steaming and discussed the fundamental processes involved in printing. Haller has investigated the reducing properties of the most common vegetable thickeners [755] and the intensifying effect produced by urea in cloth printing [754].

3. Properties of Dyed Materials.—Section **IV**-*L* should be consulted for references to the measurement and classification of color and shade by tristimulus and other methods.

Taylor and Pracejus [805] have discussed the light-fading of colored materials and have found that fading of textiles is primarily caused by radiant energy in the visible range. Sakurai [794] correlated the absorption spectra of dyes with their light-fastness. The influence of dye substituents on light-fastness has been investigated by Pinte and Millet [783]. Niederhauser [779] compared fading in the Fadeometer and in sunlight and has confirmed that they may differ. Niederhauser [780] also studied the effect of copper salts on the fastness of vat dyes. Holderegger [756] has investigated the wash-fastness of dyes.

Degradation in printing was studied by Pinte, Pierret, and Rochas [784], who followed the degradation of rayon by reducing agents, and by Schönberger [797], who studied oxidative damage catalyzed by salts of metals, such as iron and copper.

I. Viscosity, Molecular Weight, and Related Studies

1. Fractionation.—The search for more effective or more rapid fractionation techniques continues. Brooks and Badger [821] have described a two-phase system which shows promise as a means of fractionating nitrocellulose by partition chromatography. The same authors [820] have demonstrated that nitrocellulose can be chromatographed on starch columns when dissolved in a mixed solvent and may be separated by this method into fractions of different molecular weight. Fractionation by nitrogen content was observed under some conditions in both methods. Billmeyer and Stockmayer [816] and Coppick, Battista, and Lytton [826] have investigated the technique of characterizing the molecular-weight distribution of a polymer by precipitating successively larger portions of aliquots of the original solution by adding larger and larger portions of a non-solvent to each aliquot. From the amount precipitated in each aliquot and the molecular weights of the precipitated and non-precipitated fractions, the distribution can be fairly well characterized. Schulz [860] has also discussed an abbreviated fractionation procedure. The problem of freeing fractionated materials from strongly held solvents has been studied by de Brouckère, Bidaine, and van der Heyden [822].

2. Viscosity, Sedimentation, and Diffusion.—The

theoretical relationship between the intrinsic viscosity of polymer solutions and the molecular weight and thermodynamic properties of the polymer continues to receive considerable attention, along with other hydrodynamic problems such as sedimentation and diffusion. Mark [845] has critically reviewed the meaning of the intrinsic viscosity of high-polymer solutions. Eirich and Riseman [830] have suggested that Huggins' hydrodynamic correction factor is related to the deformability of a dissolved molecule and its resistance to solvent penetration. Riseman and Kirkwood [852] have developed the theory for solutions of rod-like macromolecules to the point where it is possible to propose a new method for determining molecular weights, based on the combination of intrinsic viscosity and rotatory diffusion-constant measurements. Peterlin [850] has also developed a theory for treating linear molecules showing partial solvent immobilization, and Riseman and Eirich [851] have compared his equations with the Kirkwood-Riseman theory. Flory and Fox [833] have also suggested a way of studying molecular configuration and thermodynamic properties from intrinsic viscosities. Kuhn and Kuhn [840, 841] have approached the problem of the hydrodynamics of statistically coiled molecules by studying large-scale wire models. The results obtained are compared to other theories. Schick and Singer [857] have considered the diffusion of macromolecules in solution and concluded that the driving force for this process is the free energy of dilution, and that the frictional coefficients in sedimentation and in diffusion are the same. Meyerhoff [847] has suggested a more convenient method for evaluating photographic records of sedimentation and diffusion which are made by Lamm's refractometric method.

Simha and coworkers [854, 862] have investigated the viscosities of very dilute high-polymer solutions and found no downward trend in the η_{sp}/c *vs.* c curve for a fractionated polystyrene. At the other extreme, Rivlin [853] has considered theoretically the flow properties of concentrated solutions. Krigbaum and Wall [839] have studied the viscosities of binary polymeric mixtures. They found both positive and negative deviations from ideality, and attempted to correlate the deviation with the properties of the components.

The preparation of suitable solutions of cellulose for viscosimetric measurement of D. P. continues to receive considerable attention. Launer and Wilson [842] and Doering [828] have suggested using metallic copper to prevent oxidation when cellulose is dissolved in cuprammonium hydroxide. McLean and Walker [846] and Cyrot [827] considered the variables affecting the use of cupriethylene diamine, and Heim [834] has suggested the use of sodium zincate solutions. A cooperative French investigation [815] found that, of four equations studied, the viscosities of cupriethylene diamine solutions of cellulose could be best fitted by Martin's equation. Alexander and Mitchell [814] have investigated rapid methods of measuring cellulose viscosity by nitration methods. Water-soluble polyelectrolytes can be studied best when the solutions contain an inorganic salt [829, 849], and such techniques may be useful with certain cellulose derivatives. Vollmert [867] has demonstrated that the degree of esterification of a polycarboxy compound (pectin) has a profound influence on its intrinsic viscosity. Huber [837] has critically investigated the TAPPI tube viscometer and its use, and suggests a number of changes in procedure. In several papers the problem of the flow of polymer solutions through capillaries has been considered [824, 848, 858, 866]. Higginbotham [836] has described a cone and plate viscometer for non-Newtonian fluids, particularly starch pastes.

Evans and Spurlin [832] have found that free carboxyl groups in ethyl cellulose, when neutralized with metals, cause an increase in viscosity, particularly in non-polar solvents. A similar "salt effect" has been studied by Malm, Tanghe, and Smith [844] with cellulose acetate solutions. Campbell and Johnson [823] have studied viscosimetrically the aging of cellulose nitrate solutions. The rapid part of the aging process was attributed to breaking weak interchain linkages, possibly like hydrogen bonds, while the slower part was believed to represent breakdown of the main-chain glucosidic bonds. Lude [843] has studied the structural viscosity of viscose and has also found a twofold phenomenon. Part of the structure is due to uncoiling of molecules, as in dilute solutions, while the other part consists of interactions peculiar to concentrated solutions. Clément and Pouradier [825] have studied the effect of the solvent on the shape of cellulose acetate molecules, using viscosity and surface-film measurements.

3. Optical Studies of High-Polymer Solutions.— Stuart and Peterlin [864, 865] have derived equations for the inter-relationships of such optical properties of high-polymer solutions as the electric, mag-

netic, and streaming birefringence, and the depolarization of scattered light. Schoenberg, Riseman, and Eirich [859] have measured extinction angles, sedimentation, and intrinsic viscosity of some polystyrene fractions to examine the Kuhn theory of streaming birefringence and to calculate rotatory diffusion constants. The data did not agree with Kuhn's theory, but it was shown that absolute molecular weights could be calculated from viscosity and rotatory diffusion constants using the theory of Kirkwood and Riseman. Blaker and Badger [817] have studied the thermodynamic interaction of nitrocellulose with various organic liquids by a light-scattering technique, while Ermolenko and Levina [831] have investigated the effect of non-solvent on the structural viscosity of cellulose acetate solutions. Brice, Halwer, and Speiser [819] have described a photometer for measuring turbidity, dissymmetry, and depolarization of dilute solutions of high-molecular-weight materials. Stuart [863] has reviewed the value of light-scattering techniques in high-polymer chemistry.

4. Miscellaneous.—Siegel, Johnson, and Mark [861] have found that the electron microscope may be useful in polymer studies for measuring very high molecular weights. Dilute solutions ($\backsim 1$ p.p.m.) were evaporated on collodion films, shadow-cast, and photographed. On the assumption that the observed particles were single molecules, calculations of molecular weight from volume and density agreed with viscosity and osmotic data. Booij and Waterman [818] have described a technique for measuring D. P. by a molecular distillation method.

Scheele has considered the flow processes in highly polymerized compounds [855], particularly their relation to elasticity [856]. Hencky [835] has discussed some mathematical principles of flow processes, and Krakauer [838] has described a method for recognizing pseudoplastics from their flow curves.

J. Deterioration of Materials

1. Microbiological Deterioration.—Wessel [925] has reviewed the problem of microbiological damage to textiles, and Horigan and Sage [886] have prepared an extensive literature survey of the subject for the Quartermaster Corps. Patent and book references are included in this survey. Another such review [869] prepared under Government auspices gives particular attention to test methods.

(*a*) Cellulose: Greathouse [885] and Siu and coworkers [919] have reported extensive researches into the mechanism by which microorganisms attack cellulose. Both conclude that substitution of the hydroxyl groups of the cellulose can produce resistance to attack. Siu and coworkers have reported on their conclusions in considerable detail [897, 913]. Husemann and Lötterle [888] and Pochon and coworkers [910] have discussed experiments which tend to show that the supermolecular structure of cellulose can influence its susceptibility to attack. This was pointed out by Siu [919], and also by Abrams [868] in a study of the initial deterioration of cellulose by mildew. McBee [899] has studied the available cultures of anaerobic thermophilic cellulolytic bacteria and found them to be very stable. Enebo [882] has found glucose, cellobiose, and some higher oligosaccharides as end-products produced by such cultures when acting on cellulose. Hungate [887] has isolated mesophilic cellulolytic bacteria from bovine rumen fluid. Most of the strains isolated produced end-products such as hydrogen, carbon dioxide, ethanol, and formic, acetic, lactic, and succinic acids. Hungate regards such bacteria as symbionts which have evolved in the host since they grow best when rumen fluid is added to the culture. Fåhraeus [883] has shown that a microorganism can act as a symbiont toward a cellulolytic microorganism in a mixed culture by using up the fatty acids present. Basu and coworkers [874] reported the isolation and identification of 27 fungal species found on jute, and have proposed [873] a multiple-culture technique for testing the rot-resistance of jute. A cellulase from tobacco plants has been isolated by Tracey [924], and Kristiansson [891] has studied the cellulases in malt and fungi.

(*b*) Wool: Reviews of the susceptibility of wool to biological attack have been published recently [870, 905], and interest in the details of this problem appears to continue. Stahl, McQue, and Siu [923] have reported studies on the nitrogen metabolism of a keratinolytic organism. The same group of workers has studied [922] the comparative resistance to attack of various keratins and modified keratins. A high degree of polymerization appeared to be most important in imparting resistance, while histological and molecular structures were without influence. Goldsmith [884], in a similar study, found regenerated protein fibers, except soybean protein fiber, to be less stable toward enzymatic digestion than wool. Puig [911] has isolated a number of proteolytic mi-

crobes from worn wool clothing, and suggests that they may be particularly active in causing degradation where mechanical wear has begun. Sen Gupta, Nigam, and Tandan [918] reported the isolation and identification of a wool-destroying fungus.

(*c*) Test Methods and Mildewproofing Agents: Siu and Mandels [920] have devised a new rapid method for determining resistance to microorganisms; the method is based on measurement of the oxygen absorbed during the growth of the organism. Sen Gupta, Nigam, and Saxena [917] have described a new method for isolating and studying microorganisms from deteriorated textiles.

Lawrence [894] has written a monograph on quaternary ammonium germicides including discussion of their physical and chemical properties, mechanism of action (as far as known), and practical aspects. The monograph contains an extensive bibliography on this subject. Knox *et al.* [889] have also discussed the action of such materials on bacteria and enzymes. Lee [895] has found that insoluble fungicides may be made washfast by resin-bonding them to the fabric. Nopitsch [906] described some results obtained with treatments of heavy metal salts of aromatic fungicides, and Atkins, Sen Gupta, and MacMillan [872] have investigated rotproofing agents suitable for the bitumen-saturated jute fabrics used as temporary airfield runways.

2. Photodegradation and Weathering.—Newsome [904] has reported the effect of certain black dyes on the photodegradation of nylon. None increased the tendering ordinarily observed, while several decreased it. Desson and Hadfield [880] agreed with most of Newsome's results except that they found chrome dyestuffs to cause rapid tendering unless treated with a reducing agent before exposure to light. Leigh [896] has suggested that rate studies might differentiate between dyestuffs in their effectiveness in protecting wool against photodegradation. Kornreich [890] and Smith [921] have reemphasized a point brought out in a previous paper of Smith's, that the photochemical changes in dyed materials are characteristic of the state of the whole system, not simply the dyestuff or the substrate. Winogradoff [926] has found that the deterioration of cellulose acetate by x-irradiation occurs with decrease in crystallinity. Dicker [881] has reviewed a number of aspects of the action of light on textiles.

Dean, Berard, and Worner [879] have reported the results of some weathering tests on bleached and unbleached cotton duck, and Maas [898] has investigated the effect of sunshine in the weathering of cotton sheeting. Maas exposed samples to the weather with some protected from sun, and some from both sun and rain. Fluidity, strength, and acidity were measured during exposure and subsequent storage. Bone and Turner [877] have performed experiments which may assist in understanding some aspects of the weathering of cellulose. They have shown that the region at the liquid surface of a cotton cloth dripping into water has an enhanced chemical activity leading to degradation in the boundary region and to the production of soluble brown reducing materials.

3. Mothproofing.—It appears from the literature that the chief interest at present in mothproofing is in technological refinement of the application and usefulness of known active agents rather than a search for new ones. Moncrieff [902] has set forth the current knowledge of the problem in a monograph, while numerous reviews [875, 876, 878, 900] dealing with the available mothproofing agents and their application have appeared in the literature. Lamb [893] and Moncrieff [901] have discussed some anomalous washfastness results obtained when certain mothproofing materials were applied with Xylene Fast Blue. Lagermalm, Philip, and Gralén [892] have observed a curious network in the excrement of the clothes-moth larva; the network is apparently produced in the intestines of the larvae.

4. Miscellaneous.—Reumuth [914] has proposed a systematic microscopic classification of types of damage which occur in washing rayon, and Phelps, Lund, and Norton [909] have followed chemical changes in wool during wear and dry-cleaning. Rath [912] and Samuelson and Ramsel [916] have studied damage to cellulose during chlorine bleaching, and Schönberger [797] has studied the catalytic oxidative degradation which can occur when printing regenerated cellulose fabrics. Murdison and Roberts [903] have studied various types of wear and damage to cotton fabrics by microscopic means, supplemented by fluidity and strength measurements.

Ohashi and Nakabayashi [907] have examined a number of fungicides for various starch pastes, and Pehrson [908] has discussed the preservation of carboxymethylcellulose solutions. Ryberg *et al.* [915] have examined the Fade-Ometer method for evaluating the resistance of wool oils to oxidation on storage, found it unsatisfactory, and have proposed a more reliable test [871].

Papers relating to methods for determining chemical damage to wool are discussed in Section **IV**-*C-2*.

K. Detergency and Surface Activity

1. Reviews.—Niven [962] has reviewed in book form the theory and application of detergents. A number of review articles on detergents [940, 951, 952, 964, 970, 974, 975, 979] have appeared in the recent literature. An extensive index of detergents and sulfonated oils by Sisley [972] has also been published.

2. Mechanism of Detergency.—Roebuck [968] has summarized present knowledge of the relation between the chemical structure of detergents and their action in reducing the adhesive forces between soil and the contaminated surface, and in solubilizing the soil. Chwala [934], in the same volume, reviewed the influence of constitution on the mode of action of anionic detergents. Gruntfest and Young [942] have made some systematic microscopic observations of the behavior of carbon particles and fibers in the presence of detergent solutions, and have extended their conclusions to the results of simulated laundry tests. Several articles [939, 949, 978, 986] have dealt with aspects of the soil substrate in evaluating detergents, or with properties of fiber which may affect the effectiveness of detergents. Such interest in the role of the soiled fibers themselves in detergency may lead to a better understanding of the process, as well as to more work such as that reported by Utermohlen [981] on methods to improve the soil-resistance of cotton cloth.

The nature of the micelles which form above certain critical concentrations in solutions of soaps and detergents has not been settled. Philippoff [965] has reviewed the available data from x-ray diffraction experiments on this problem and concludes that small, laminar micelles are most likely. However, Hughes [945] has disagreed with some of Philippoff's interpretations of x-ray evidence. Marsden and McBain [955] have reported some new data on triethanolamine laurate solutions, and Klevens [947] has stated that the addition of hydrocarbon to soap solutions causes only a slight decrease in the critical concentration for micelle formation. Hoyer and Mysels [944] have suggested a method for studying the properties of micelles which involves tagging the micelles with small amounts of dye. Aniansson and Lamm [927] have been able to measure the adsorption of octadecyl sulfate in liquid surfaces by labelling the detergent with sulfur 35. Thermodynamic studies of soap solutions, assuming spherical micelles, have been made by Stainsby and Alexander [976], and experiment was shown to agree with their theoretical considerations. Vold and Konecny [984] have presented some evidence that the ability to suspend powders is not related to potential detergent power. Doscher [936] has reported on some systems where carbon suspendability is not related to zeta-potential, although the latter has been suggested as an important factor in detergency. However, Lindner [953] has proposed a soot-suspension technique to evaluate the protective colloid action of detergents, and Weatherburn and coworkers [987, 988] have measured the suspending power of soaps and built soaps for carbon black and other materials. Weatherburn found that alkaline builders decreased the suspending power of soap, but he has also shown [931] that the addition of carboxymethylcellulose to built soap can almost compensate for the decreased suspendability.

Vaughn and Smith [982] and Merrill and Getty [956] have examined the detergent properties of alkyl aryl sulfonates when used with various builders. Further evidence of the usefulness of carboxymethylcellulose in builders has been presented by Vaughn and Smith. Dicker [935] has also reviewed this subject and has given some experimental results. Morrison and Campbell [958] have considered the emulsifying power of water-soluble ethers of cellulose and have found that mixtures of sodium carboxymethylcellulose with other soluble ethers produce more stable emulsion than the former alone. Kubias [948] has shown that the relative effectiveness of polymetaphosphates as builders may be determined by the use of Schwarzenbach complexes, and Reutenauer [967] has studied the wetting and emulsifying powers and resistance to hard water of mixtures of detergents with potassium soap.

3. Testing.—Snell [973] and Marroc and Sisley [954] have given fairly comprehensive surveys of the present situation in detergent testing. In the latter paper there is a brief description of Marroc's new laboratory washing machine, the "Lavatomic," together with some results obtained with it. Lambert and Sanders [950] have pointed out some physical-chemical limitations of present test methods, and have proposed [969] a test which attempts to duplicate service conditions more closely than many laboratory tests. Vaughn and Suter [983] have reported on a detergency test which measures soil-removal by

measuring the transmission of the detergent solution, and whiteness-retention by the reflectance change of unsoiled swatches agitated in soil-detergent mixtures. The results were correlated with commercial laundry trials. Barker and Kern [929] have compared measurements of soil-removal made by determining the optical transmission of the wash liquor with reflectance measurements on the washed sample. These do not always agree for a given detergent, and the authors have proposed a criterion for determining when the transmission method may be expected to give reliable results. Harris and Brown [943] have discussed the complicated problem of preparing standard soiled fabrics, and Draves and Sherburne [938] have reported the details of preparing the printed soil cloth developed at the instance of the U. S. Bureau of Ships. Nutting [963] has described the final model of the A.A.T.C.C. "Detergency Comparator" which has been designed to evaluate detergents under conditions of high concentration and low bath ratio such as is frequently found in textile processing. The A.A.T.C.C. committee on wool scouring [928] has presented a tentative method for evaluating the continuous scouring of grease wool.

Bartholomé and Schäfer [930] have developed a theoretical equation for the change of surface tension with time, which is an important aspect of the wetting process. The rate of surface-tension lowering has been investigated by Burcik [933] for solutions of sodium laurate, sodium dodecyl sulfate, and sodium oleate. By assuming that the change is determined by diffusion of surface-active molecules from the interior of the solution, Burcik was led to an equation which agreed well with surface tension and patch-sinking measurements. Shapiro [971] has reported experiments which show that woven tapes have advantages in speed, convenience, and reproducibility over usual skein samples used in the Draves test. Measurements of the rate of wetting were also described. Trommer [980] has suggested a test method in which wetting and penetration can be studied separately. Blumenstein [932] has examined the A.A.T.C.C. rewetting test and has suggested that a higher temperature of impregnation would give results more in line with plant performance.

Several analytical techniques for detergents and builders have been described in the past year. A scheme of analysis for commercial detergents has been suggested by Gilby and Hodgson [941]. Draper and Milligan [937] have investigated the Karl Fischer method for water in soaps and synthetic detergents. The estimation of anionic detergents in solution by the Hartley titration procedure has been studied by Swanston and Palmer [977]. Wallin [985] and Karush and Sonenberg [946] have proposed colorimetric techniques for determining sulfated or sulfonated surface-active agents, while Reutenauer [966] has studied the determination of the usual inorganic salts found with such materials. Neu has published a series of papers on the analysis of washing and cleansing agents, including such subjects as the titrimetric determination of orthophosphoric acid [959], use of base exchanger in measuring acid number and equivalent weight [960], and differentiation between ethers and ether glycolic acids of cellulose [961]. Miller [957] has described a colorimetric method for determining water-soluble silicates in detergents.

L. Equipment and Techniques

In the previous survey of this field [32], it was stated that the trend in 1949 appeared to be toward the development of new and improved instruments for the control of processing operations in industry and for the examination of finished textile materials. These newer methods have been used effectively during 1950 for the evaluation of the older and more conventional practices of control. More emphasis was placed on this part of the field of equipment and techniques than on the development of new methods, although a considerable amount of work on instrument improvement was undertaken in 1950.

1. Test Instruments and Methods.—Hünlich [1017], Krassowsky [1024], and Matthes [1028] have reviewed general aspects of textile testing, and a second edition of Skinkle's book on this subject has appeared [1043]. Lomax [1026] has reviewed the changes in textile testing in Great Britain over the last 14 years; and, in handbooks published by the Ministry of Supply [922] and the British Standards Institution [990], current British practice has been reported. Schwen [1040] has reported on some experiments toward the standardization of test methods for textile assistants, and problems concerned with tests and specifications for felt have been discussed by Stevens [1045]. Cuckler [999] has described the development of special equipment for basic studies on textile materials at Textile Research Institute.

The large number of papers on mechanical meas-

urements of single fibers and filaments illustrates the growing interest and importance of this phase of textile testing. These papers are concerned with the physical properties of rayon filaments [1003], and the tensional [1031, 1032] and bending [1030] characteristics of single fibers. Kawai and Tokita [1020], Pinoir and Dubois [1036], and Sinclair [1042] have described different methods for measuring the Young's modulus of fibers and films. A number of methods for determining the physical properties of textile fibers by force-elongation studies are of interest [1000, 1007, 1027]. Wegener has reported on a new stress-strain tester for dynamic endurance [1048, 1049]. Lohmann has described an electronic recording analytical balance [1025] which is free of mechanical connections with the balance beam, is direct-reading, sensitive, and rapid; a torsion creep unit of high sensitivity has been developed by Johnson [1019]. Hogan has devised a nomograph for calculating the degree of elongation of a fiber during its preparation [1013]. Punched-card techniques have been applied to the analysis of data on the physical properties of single fibers [1047].

The sensitive equipment developed for the study of single fibers has been applied to the testing of roving, sliver, and top. The little published information on this field [995, 998] is probably not indicative of its potentialities. New methods and equipment for the testing of yarn strength [993, 1001, 1004, 1008, 1010, 1050] have been discussed; yarn-tension equipment developed by the General Electric Company [994], American Viscose Corporation [989], and Zellweger [1011] have been described.

In the field of fabric testing, Painter, Chu, and Morgan [1035] have studied the tear-resistance of fabrics on the Elmendorf Tear Tester. Dimensional change in woven fabrics has been measured on the "Strip-Tester" [991], and Schefferle [1038] has reported on the testing of fabrics for strength and has described the pendulum tester, the burst tester, and constant rate of loading equipment. For further detail on the relationships between fiber, yarn, and fabric characteristics, the reader is referred to Section **IV**-*F* of this review.

Considerable attention has been given to the measurement of the "hand" [996, 1034], drape [997], and stiffness [1029] of fabrics and to the broad fields of crease-resistance and abrasion- or wear-resistance. A comprehensive survey of the patent and published literature on creasing and crease-resistance of textiles has been prepared by the Quartermaster Research and Development Laboratories of the Department of the Army [1015]. In addition to this survey, Hebeler and Kolb [1006], Koch and Bohme [1023], and Quehl [1037] have reported on studies in the field of fabric wrinkling and creasing. Köb [1021] has made measurements to attempt a determination of crimp in cotton fabrics. Developments in Germany on the wear-resistance of textiles during World War II have been summarized in a collection of papers translated and compiled at the request of the Department of the Army [1033]. The Research and Development Laboratories of the Quartermaster Corps have also provided a literature survey for the years 1936–1949 on the serviceability of fabrics [1016].

Various aspects of testing with the "Taber Abraser" have been reviewed by Zook in a series of articles in the *American Dyestuff Reporter* [1051–1054]. Jackson [1018] and Holderer [1014] have considered some general factors in the abrasion and wear characteristics of textile materials. Fletcher and Gilliam [601] have investigated the abrasion-resistance of rayon fabrics, while Germans [1002] and Schiefer and Krasny [1039] have studied the abrasion of wool fabrics. A new abrasion test method has been described by Koch and Bobeth [1022]. The use of nondurable aluminum salt-wax emulsions to increase wear-resistance of fabrics is shown to be unwarranted where garments are subjected to severe laundering and service conditions [1046]. The abrasion-resistance of sized yarns has been investigated by Hildebrand and Parriaud [1012] and by Stallings and Worth [1044]. Hayek and Chromey [1005] have discussed the measurement of static electrictiy on fabrics, and Henno and Jouhet [1009], the measurement of the dust-retention of fabrics. Silverman and Viles [1041] have reported on the determination of cotton textile dusts in the air.

2. Control Instruments and Methods.—In textile manufacturing, the examination and grading of material starts with the fibers as received in the bale [1135, 1169]. In this examination fiber length and fineness are of primary importance. Reports on the theory of random sampling [1055] and definitions of fiber measurements [1142, 1153] have been made by a number of European authors, and the limitations of air-flow methods in fiber-fineness determination have been discussed [1086, 1089, 1097, 1147, 1150]. Comparisons of the air-permeability method with other types of fineness measurements have also

been made [1111, 1140]. A modified technique for determining cotton maturity has been reported by Carpenter and Lee [1090]. The use of gradient tubes in fiber-density studies [1141] has been suggested. Theoretical and experimental treatments of the application of the Kozeny equation to consolidated porous media are of interest in fiber studies [1056, 1173].

Methods of measuring the uniformity of roving and yarn [1129, 1139] have been compared and reviewed, and several uniformity instruments [1058, 1105, 1149, 1162] have been described in detail. In addition, numerous accounts have been given of the evaluation and importance of uniformity measurements as well as the handling and use of uniformity data [1063, 1081, 1096, 1102, 1143, 1161, 1167].

It is encouraging to observe the increased use of statistical methods in the textile industry for the analysis of control and test data. Papers in this field include discussions of the theory of statistical analysis [1078, 1085, 1108, 1110, 1113, 1122, 1158] as well as the application of statistical methods to specific problems [1075, 1080, 1109, 1148, 1156]. Hessler and Kieke [1118] have reported details of the use of a fiber blender for blending samples of bale cotton for testing. Two tests of the spinning quality of cotton have been proposed by Dunkerley [1101] and by Gupte, Navkal, and Sen [1114], who have described a test using only 60 g. of cotton. Equipment has been developed for the continuous determination of the mechanical characteristics of a yarn [1116] and the automatic recording of end-breakages [1059]. The use of stroboscopic techniques [1146] and high-speed photography [1103, 1157] in textile production has been reviewed.

The control of relative humidity and the determination of the moisture content in textile materials have received active attention. Equipment for the measurement and control of humidity [1088, 1095, 1104, 1133, 1159, 1166] is under development and is particularly important since the effects of moisture on the properties of textile materials are not clearly understood. The evaluation of various electronic meters for determining the moisture content of textiles has been continued [1120, 1126, 1127, 1168]; and investigations of other instruments and methods for moisture determination in textile materials have also been reported [1092, 1130, 1160]. The nuclear magnetic resonance absorption technique [1151] is reported as a promising method for determining moisture content. Methods for measuring the dimensional changes of yarns [1067, 1115, 1117, 1165] have also been described.

The influence of pH in textile manufacture is of great importance and, accordingly, much attention has been given to the exact control of pH during various processes. Chaplin [1093] has considered some of the factors affecting regulation of pH in industry, giving a brief introduction to the theoretical aspects of pH control with particular reference to control-rate characteristics, buffer index, acidity characteristics, and equilibrium lag. Mooradian and Millson [1132] have discussed the importance of pH control in the processing of wool. Lister [1124] has conducted further investigations on the pH control of acid and chrome dye baths. Stearns [1155] and Skinkle [1152] have made contributions to the quantitative evaluation of dye strengths, dye characteristics, and dyeing methods; while Wright [1172] has reported on the minimum difference in colors which is necessary for discrimination between them. Applications of the automatic tristimulus integrator to textile-mill practice have been described by Davidson and Godlove [1099]; the use of this integrator with the General Electric recording spectrophotometer has been discussed by Davidson and Imm [1100]. Barr [1076] has emphasized the importance of time and temperature control in earlier processing in connection with felt dyeing. Plans for automatic process control have been reviewed, together with a discussion of a number of instruments used for such purposes [1070, 1072, 1079]. Moncrieff [1131] has discussed dye-bath control equipment, and Walter [1163, 1164] has pointed out the improvements in economy and uniformity resulting from the use of automatic instruments. Other papers on equipment include discussions of instrument maintenance [1134] and the use of viscometers [1084] and rotameters [1119].

A large number of the publications in this division concern the inspection, examination, and testing of the finished product—dyed fabrics. These articles include discussions of the control of dye baths [1171]; testing the dyeing properties of various dyes [1071, 1094, 1128]; the measurement of tippiness in wool staples [1112]; cloth inspection machines [1060, 1068]; automatic devices for the comparison of luster of textiles [1061], and for the measurement of color and color changes [1062, 1064, 1066, 1074, 1077, 1098, 1106, 1138]; color systems and notations

[1083, 1136, 1137]; theoretical analysis of the measurement of color [1087], gloss [1144], whiteness [1073], and color matching in general [1057]; the uniformity of grading of lightfastness standards [1069, 1107]; testing lightfastness [1065, 1091, 1145, 1174] and perspiration colorfastness [1125]; and flammability measurements on thin sheet materials and fabrics [1082]. Also of interest are the various papers on water-repellency of fabrics [1121, 1123, 1154, 1170].

3. Identification and Examination.—The various reports on methods of identifying and separating textile fibers and fiber mixtures are both numerous and diversified [1178, 1187, 1188, 1191, 1196, 1197, 1203, 1213]. Chromatographic techniques have had wide application to problems in textile research and development [1181, 1183, 1186, 1192, 1195, 1202, 1212, 1214]. Phase-contrast microscopy, a relatively old technique, has found increasing application to the study of textiles [1185, 1198, 1199] and augments the conventional methods of microscopy [1179, 1193, 1200, 1201, 1208, 1210, 1211]. Techniques using the electron microscope [1182, 1190, 1194], radiation from radioactive substances [1175–1177, 1180, 1184, 1205, 1206], and x-ray diffraction analysis [1189, 1204] have also been applied to the study of textile materials, especially in problems related to morphology and structure. Fluorescent materials have been used as tracers in the felting process [1209]. Microwave techniques in the 3–10 cm. wave-length region have been applied to the determination of the dielectric properties of fibers and films of high polymers [1207].

V. Development

The papers in this section have been divided into three groups which are arranged primarily according to processing operations and in the order that these processes occur in manufacture. The first section, on manufacturing methods, deals with the production of grey goods. The second section is concerned with the wet-processing methods used in converting grey goods into finished materials. The third section contains miscellaneous subjects, such as instrumentation, waste disposal, etc. In a few instances, however, interest in a specific problem has warranted deviation from this general outline.

A. Manufacturing Methods

1. General Processing.—The use of fiber blends and the adaptation of machinery, designed originally for one specific fiber, to the production of blended yarns and fabrics have been the subject of much investigation. Whiffen [1236] has reviewed the development of modified worsted processes, especially the changes in procedure made possible by high-draft spinning and the reduction in the number of operations through the introduction of new types of machinery at various stages. Heathcote [1224] has analyzed the process of manufacture of worsted yarn on the American system, including data on the efficiency and waste in this process. The factors which are important in the production of staple yarns on worsted ring frames [1215, 1220] and on the cotton system [1218] include spindle speed, humidity, temperature, doffing time, and type of fiber. The effect of manufacturing processes on fiber-length distribution and staple length of raw cotton has been discussed by Williams and coworkers [1238]. Radcliffe [1234] has published a book on *Woollen and Worsted Yarn Manufacture.*

General reports on the processing of nylon have been given by Millard [1231], Garner [1222], Marsden [1230], and others [1216]. The processing of Dynel on the cotton system has been discussed by Gaines and Stowell [1221]. Experience in the development and use of the Pacific Converter has been reviewed by Kennedy [1227] and Wilkie [1237]. There appears to be a marked increase of interest in machines of this type. Nordahl [1232] has contributed an illustrated account of the production of glass filament fabrics.

Little [1229] has presented a popular review of contemporary knowledge about static electrification in the textile industry. In particular, he has discussed the influence of static generation on textile processing and methods whereby such electrical effects may be reduced. Taylor [1235] has suggested five means for reducing the undesirable effects of electrical-charge generation in yarns. These include the lubrication of yarn, the incorporation of suitable conductors in the lubricant, the maintenance of a fairly high relative humidity, the proper grounding of equipment, and the use of suitable ionizing devices to increase the conduction of electricity in the air.

Keggin, Morris, and Yuill [1226] have measured the quantity of charge developed on wool, casein, Ardil, nylon, and cellulose acetate fibers as a function of relative humidity. They have found that the quantity of charge decreases as relative humidity increases but levels off at about 60% to 70% R.H.

Cotton, flax, and viscose do not show this tendency to level off. Gérard [1223] has investigated the origin of static electricity during the processing of staple rayon and has discussed methods for eliminating static. Steam treatment has been suggested as a means of elimination of static on Peralta rollers used in connection with wool carding [1217].

Cotterill [1219] has investigated the effects of humidity on rayon yarn and has made recommendations about suitable conditions for the storage and manipulation of various rayons. Pollitt [1233] has discussed the effect of humidity on the phyiscal properties of various types of yarn, and Ingham [1225] has emphasized the importance of humidity in the processing of wool fibers. Electrostatic phenomena in wool processing have been examined by Lion [1228], who has outlined a practical research program for the study of methods to overcome the difficulties encountered in fiber electrification.

2. Opening and Blending.—The increasing use of synthetic and natural fiber blends has led to the development of several new systems of opening and blending. Anderson [1239], Leineweber [1246], Helliwell [1244], and others [1242] have described various systems for processing blends. The most significant trend in opening and blending has been toward the development of fully automatic blending machinery. A discussion of the Benoit and Platt systems, the automatic system of Proctor and Schwartz, and the Oddie-Radcliffe continuous system has been presented [1240]. The Spencer-Halstead and other systems have also been described in detail [1241]. Various methods of worsted color-blending have been reviewed by Beevers [1243].

The Southern Regional Research Laboratory [1245] has developed a new cotton opener which is especially useful in opening mechanically-harvested cotton. The opener produces less neps and has a higher capacity than conventional openers. A new arrangement of cleaning and blending machinery at Columbus Manufacturing Co. is reported [1247] to result in cleaner and evener picker laps.

3. Scouring and Milling of Wool.—Loss in quality quite often follows the acceleration of various wet processes in woolen and worsted manufacture. Noble [1264] has discussed means for reducing the time of wet processing without lowering the quality of the finished goods. Whewell [1272] has stressed the importance of correct scouring in properly preparing fabrics for dyeing and finishing. Reviews of wool scouring show a definite trend toward the use of synthetic detergents [1253, 1255, 1263, 1268]. La-Fleur [1259] has compared the effectiveness of the detergent action of various types of surface-active products on wool cloths soiled with oil. Neutral scouring with polyoxyethylene thioethers, using sodium chloride, was found to be superior to all other methods examined. The effect of pH, salt concentration, temperature, and other properties on the relationship between detergency and absorption by the fiber has been studied by McLaren [1262].

Stadler has traced the development of wool detergents, particularly those for use in acid media [1270]. The essential conditions which should be observed when scouring wool with detergents have been summarized by Hepworth [1256]. His experimental work indicated that for most economical scouring with these materials the addition of both soda and a salt builder was necessary. It appears [1248] that, in scouring raw wool with detergents, waste disposal becomes a particularly difficult problem. New methods have been developed for studying detergent power [1261, 1265] and for determining wool fat content [1254, 1267]. Hoffman [1257] has described a dry-cleaning process for degreasing wool; his method is reported to increase the yield of tops, to decrease card waste, and to improve the quality of goods so that less expensive blends can be used. It has been shown experimentally [1249] that wool may be scoured by ultrasonic techniques in only 15 sec. There have been several reports [1250, 1260, 1271] on scourable sheep branding fluids which should help alleviate one problem in wool scouring. Bryant [1252] has proposed a foam method for degumming raw silk which results in less filament breakage in subsequent reeling and coning.

Harris and coworkers [1251] have developed a simple quantitative test for evaluating the felting of wool top and have applied it [1269] to a number of wools of different geographical origin. Only very small differences were found in the felting of these wools. Peryman and Speakman [1266] have discovered that the influence of lubrication on the felting and milling of wool is apparently considerably greater than has previously been supposed. Hückel [1258] has discussed the formation of hair felts and the influence of various milling agents on felting.

4. Carding.—For many years there have been no significant developments in the body of wool or cotton cards. Marsden [1281] has prepared a list of the

major recent developments in wool carding, which appear to be few in number except for the application of more suitable materials, such as aluminum and steel, for structural fabrication. Much effort has been expended, however, for a better understanding of carding machinery [1289]. The general principles of wool carding have been explained by Marshall [1282], Shaw [1284], and Varga [1286, 1287]. Townend [1285] and Hind [1279] have studied the interaction of worker and swift on the woolen card. Martindale [1283] has supplemented this work with an investigation of the efficiency of the fancy and doffer in clearing the swift under various conditions of operation. The addition of another worker, as suggested by Gilljam [1277], is reported to increase the output of the card. According to Crofts [1276], the Peralta machine, which crushes all foreign matter, has made possible some improvements in yarn strength. Choice, merits, and maintenance of card clothing have been considered [1274], together with recommendations for settings and speeds of various parts of the carding set.

There have been few developments in the carding of cotton. Bogdan [1275] has presented an excellent review of the literature on neps in cotton. Loveless [1280] has reported on work in progress on the cause and measurement of neppiness, and Gulati [1278] has reported extensive studies on these problems. Wegener [1288] has compared two methods of feeding cards on the basis of the uniformity of sliver produced. He has concluded that oblique lap joints show 25% to 35% lower fluctuations in sliver count than joints runnning parallel to the feed rollers. Considerably improved efficiency has been claimed for a new continuous cylinder stripping motion [1273].

5. Drafting and Spinning.—

(*a*) Drafting: Efforts to maintain and increase the uniformity of textile materials and simultaneously to increase the productivity of textile machinery have led many workers to study the theoretical and practical aspects of the drafting process. Zotikov [1329, 1330] has examined in a theoretical way the forces which operate on sliver during drafting, taking into consideration pressure on the rollers, diameter of the rollers, and thickness and width of the sliver. A method for measuring these forces has been described. Draft as a means of fiber parallelization has been compared with carding and combing and proves to be more effective. The Ambler high-draft system [1290] is an important example of a new process developed on the basis of a mathematical theory of drafting [1303]. Champion [1298], Graham [1301], and Fahrbach [1299] have also presented analyses of the drafting process in spinning. A chart has been presented by Bogdan [1295] which shows the necessary correction in yarn count due to twisting of roving. Löfgren [1306] has studied the influence of fiber length and distribution on the working progress in Paul's draw field. The distribution of fibers in rovings and roving sections has been investigated by Köb [1305].

The formation of neps during the multiple drawing of cotton has been considered by Loveless [1307]; as the number of drawings increased, the strength, neps, and uniformity increased while the elongation decreased. Morton and Summers [1310] have described the behavior of Fibro slivers in the drawframe. Fiber breakage in worsted drawing and combing has been dealt with by Townend [1326] and Griffin [1302]. Popp [1317] has described a high-draft head for fly-frames.

(*b*) Spinning: An excellent book by Morton [1311] on the subject of spinning has recently been published. Boult [1296] has reviewed the modern developments in throwing machinery. Studies by Baltz [1293], Reinfeld [1320], and Stein [1322] of the kinematics of the ring traveller should be of particular interest to machinery manufacturers and users, together with two theses by Newell [1313] and Taylor [1323], which are concerned with lubrication of spindles.

Problems connected with the development of efficient woolen-fiber lubricants have received attention by Martin [1308]. Mühle [1312] has studied the conditions a mineral-oil lubricant must meet in order to be a satisfactory substitute for olein. Townend and Yu [1327] reported that end-breakage is reduced in worsted drawing and spinning through the use of anti-slip agents, such as colloidal silica, in the lubricant.

Excerpts from a talk by Farnsworth [1300] have given a description of the centrifugal pot-spinning frame that is being operated experimentally in England to produce worsted yarn. Ullrich [1328] has described a German ring spinning machine designed to spin wool sliver. Newell [1314] has outlined the Bird universal drafting system, and some details of the Hegemax spinning process have been given by Batorffy [1294]. The Hegemax is a device

for imparting a preliminary twist in ring spinning so that the yarn is strengthened before ballooning. New machines and drafting systems from H & B [1309] and McGlynn Hays [1291] have also been discussed. Ross [1321] and Porsche [1318] have considered the value of shortened spinning processes to achieve economy in labor, the abolishment of redundant operations, and machinery improvement.

Permanyer [1316] has investigated the effect of twist and tension on yarn quality, while Johannsen [1304] has given a comprehensive analysis of fiber breakage on the ring spinning machine. Temmerman and Hermanne [1324] have demonstrated the application to the cotton system of an absolute measure of irregularities which is independent of the count, and have shown how it changes throughout the spinning process. Oertel [1315] has conducted a series of spinning tests designed to study mechanical factors relating to yarn-tension regulation in the spinning of fine yarns at high speeds. The number of yarn breaks as a criterion for the design of drafting heads has been discussed by Toenniessen [1325], who concluded that the lea break shows greater accuracy even though it requires much less time to carry out. Pringle [1319] has outlined the theory of flax spinning, and Caldwell [1297] has published information on the proper flax spinning frame settings for maximum output. A comparison of the English and French systems of worsted drawing and spinning [1292] has shown that the English or Bradford system is best suited to wools of staple length 4 in. and longer, while the French system utilizes short tender wools which are spun to soft-textured yarns.

6. Winding.—Inderfurth [1337] has outlined some considerations which are important in maintaining the quality of winding of synthetic yarns. This subject has also been discussed by Hulme [1336] in a paper on the preparation of continuous-filament yarns for weaving. Considerable interest continues to be shown in machinery for high-speed precision winding [1333, 1334] and warping and beaming [1331, 1332]. Emmerich [1335] has investigated the possibility of obtaining constant cop weights in doubling-room operation.

7. Sizing and Desizing.—

(*a*) Sizing: Probably no other process in the manufacture of grey fabrics has received as much attention through the years as sizing. While starch continues to occupy an important place in this operation, materials such as synthetic high polymers and cellulose derivatives are becoming increasingly important. Jensen [1347] has discussed some general aspects of the problem of evaluating sizing. The properties of starches and modified starches with particular reference to practical sizing problems have been reviewed by McGill [1351]. Jensen has studied the characteristics of starch pastes and films containing varying amounts of tallow [1348], and the effect of various additives on the adhesiveness of starch pastes [1349]. Foulon [1344] has described the use of carboxymethylcellulose for sizing, both as an additive to starch sizes and alone; Ulrici [1357] has indicated that alone it may have important advantages over starch. Möller [1352] has described the starch ether derivatives called Solvitoses, which do not show retrogradation and may, therefore, be useful for some applications where this phenomenon causes trouble with starch itself. It has been reported by DuPont [1340] that the most satisfactory size for raw nylon is polymethacrylic acid, but that polyvinyl alcohol may be used when the corrosion-resistant equipment necessary for the former is not available. Weaver [1359] has concluded that the quantities of sizes now used on continuous-filament rayon yarns may be too small and that solution of this problem may be found by coating each thread with a thick layer of size immediately upon spinning. The single-end sizing of cotton yarns at the spinning frames has been studied by Hill [1345], who believes that yarns of good weaving qualities could be produced by such an arrangement.

New trends in slasher controls include the automatic regulation of slasher speed, of squeeze roll loading, and of water level, and the continuous recording of size viscosity [1338]. Prechter [1354] has described the use of multi-motor slasher drives which permit close control over yarn tension at all points within the slasher, and Campbell [1342] has described a drive which utilizes a magnetic clutch. Descriptions of the Gaulin homogenizer and the advantages of homogenizing warp size have appeared [1339, 1343]. Small-scale slashers for experimental purposes have been produced by Callaway and Monsanto [1341].

(*b*) Desizing: Pfannmuller and Noe [1353] have reviewed the application and evaluation of enzymes in desizing, and Marston [1350] has more briefly discussed this subject. The effect of several wetting agents on enzymatic desizing has been studied by Jensen [1346], and Voss [1358] and Schubert

[1355] have discussed problems in evaluating the effectiveness of enzyme preparations. Schubert has also discussed [1356] the practical importance of the chemical properties of enzymes in their stability and activity.

8. Weaving.—During the past year much of the literature on weaving has dealt with loom developments. Snowden [1379] has described a number of automatic looms for fancy woolen and worsted weaving, and has also discussed [1381] some general weaving problems. A new principle of loom design wherein the reed remains stationary at all times and the fell of the warp is brought to the reed has been outlined by Hart [1369]. Honegger [1374] has reviewed the recent achievements in Swiss loom manufacture, and Thomas [1382] has contributed a three-part article discussing developments in power looms. The latter work has been supplemented by studies of the various motions on the looom as affected by higher loom speeds [1361]. The operation of the Fayolle-Ancet circular loom has been described in some detail [1360]. A new British loom [1364] features simplicity of design and interchangeability of parts. A novel multi-sectional loom with centrifugal weft supply [1363] has been developed which combines the characteristics of circular and orthodox looms. The Ballbe shuttleless loom has also been discussed [1362]. Gunn [1368] has reported the advantages obtained by using a right-hand filling stop motion and has shown how this motion may be fitted to Draper E model looms. Demartini and Lukens [1366] have outlined a method for calculating requirements of the drive for a loom from a knowledge of system constants and without the necessity for extensive testing.

Snowden [1380] and Hollstein [1373] have studied the problem of warp tension in weaving. The measurement of this tension and the effect it has on fabric properties have been discussed in detail. Henno and Jouhet [1370] have also outlined a method for measuring the tension of warp yarns. An instrument for measuring shuttle tension has been described by Saxl [1377], and should aid in preventing the many fabric defects improper shuttle tension can cause. A comprehensive review of defects in fabrics has been prepared by Goldberg [1367]. Laird [1376] has outlined the effect of uneven spacing of reed wires. Schneider [1378] has shown the correlation between shed formation and sley motion, and has determined the maximum possible number of picks for the various heights of the shed in a number of Jacquard systems. Hesse [1371] has attempted to clear up the question of the practical utility of curved sley in weaving, and has also summarized [1372] the practical experience gained in the weaving of rayon staple and has concluded that rules and principles applicable to cotton weaving hold in the weaving of rayon.

Coke [1365] has reviewed the production and properties of non-woven fabrics, while Kershaw [1375] has described the British bonded fabric known as "Supatex."

9. Knitting.—Lancashire [1389] has prepared a comprehensive review of progress in knitting-machine manufacture from 1936 to 1950. In a systematic investigation of the basic loop interlacings of plain knitting machines, Henning [1386] has presented the possibilities for application and further development of these interlacings in designing. Reichman [1391] has considered the proper storage and handling of knitting yarns. Paling [1390] has reviewed the technology of warp knitting, and procedures in the warp knitting of nylon have also been discussed [1383]. The advantages and disadvantages of single-head and multi-head machines for hosiery production have been summarized by Durrand and coworkers [1384]. Heckert [1385] has briefly explained the use of nylon staple in hosiery, while Lancashire [1387] has given an account of a unit for feeding elastic rubber yarn to hosiery machines. Lancashire [1388] has also described the manufacture of knitted plush.

B. Wet Processing

1. Dyeing and Printing.—

(*a*) General: Fargher, Taylor, and Thomas [1426] have discussed the way in which yarn irregularities introduced during spinning can affect dyeing and finishing processes. The limitations which fabric tension places on the usefulness of jigs in dyeing and scouring have been explained by Hall [1436]. Marr [1462] has reviewed open-warp dyeing machines and the pressure dyeing of warp yarn. Procedures for dyeing with Coprantine colors, which are azo dyestuffs requiring aftertreatment with copper salts, have been outlined by Stern [1490], while Luttringhaus [1459] has described the merits of dyestuffs which must be metalized by aftertreatment with chromium. The properties, method of application, and advantages of a cation-active after-

treating agent for substantive dyes on cellulosic fibers have been presented by Weber [1501]. Gund [1433] has given details of a new Indanthrene Blue which is claimed to be distinguished by the clearness of its shade and its behavior in the presence of caustic soda. Heyder and Sandor [1441] have compared the vat-acid process with the pigment-pad, indigosol, and conventional vat dyeing processes, and have shown that each process has advantages in specific cases. Celentano [1414, 1415] and Nuttall [1473] have outlined the techniques for successful naphthol dyeing, and Kirst [1452], Hees [1439], and Giesen [1432] have given additional attention to particular aspects of this procedure.

(*b*) Blends and Synthetics: The dyeing of new hydrophobic fibers continues to be one of the most difficult of the colorist's current problems. Douglas [1419] and Schläppi [1484] have reviewed the general aspects of the problem of dyeing nylon. Allison [1392] has described the chrome-steam process for dyeing this fiber, while Niederhauser [1471, 1472] has investigated the use of acid dyestuffs. Carter and coworkers [1412] have found that nylon can be mordanted with tannic acid and can then be dyed by vat dyes at normal temperatures. Vat dyeings on tannic acid-treated nylon, although not as fast to light, washing, and rubbing as on cotton, give better results in all these respects than other types of dyestuffs. Früh [1431] has examined the application of most classes of dyestuffs to the German caprolactam polyamide fiber, Perlon L. Most dyestuffs can be applied but with varying degrees of success. According to Köster [1455], the dispersed acetate dyestuffs are most suitable for the polyurethane fiber, Perlon U. Müller [1469] has discussed the effect of various processing conditions on the dyeing of these two Perlon fibers. Reports have been made on papers presented before the A.A.T.C.C. by Feild [1427, 1428] on the dyeing of Dynel, and Meunier [1465, 1466] on the dyeing of Orlon and Fiber V.

The use of nylon in mixtures with other fibers, particularly wool, presents an especially difficult dyeing problem, which has been discussed by Burnthall [1409], Hees [1440], Smith [1488], and Thummel [1497]. There have also been reports of useful information on the dyeing of other blends, such as wool and cellulosic fibers [1482, 1502, 1503], wool and cellulose acetate [1394], and cellulose acetate and viscose [1396, 1464, 1470].

Tyler [1499] has described some problems in dyeing rayon, and Campbell [1410] has investigated the difficulties encountered in trying to dye rayon with direct cotton dyes when only hard water is available for processing. York [1504] and Baumann [1402] have discussed the dyeing and printing of cellulose acetate fabrics, while Hilliard [1442] has outlined some practical problems involved in the dyeing and finishing of acetate warp knit fabrics.

(*c*) Wool: According to Luttringhaus [1458], the current trend in the wool industry is toward increased use of dyestuffs with which improvements on one or more of the following factors can be obtained: penetration, uniformity of coverage, fastness to light, fastness to fulling, fastness to peroxide bleaching, length of dyeing cycle, or preservation of wool fiber. Metallized acid dyes, leuco-esters of vat dyes, and Helindon vat dyes make these improvements possible. Joerger has written an extensive series of articles on the dyeing of wool stock, tops, and yarn, covering equipment and technique [1444], the use of acid dyes [1445], chrome dyeing [1446, 1447], and the use of vat colors [1448, 1449]. The application of vat dyes to wool appears to be of increasing importance. Cheetham [1416] has discussed their use on wool and wool-rayon mixtures with additives which prevent excessive alkali damage to the wool and improve levelness in the mixed fabric. The use of vat dyes of the Indigosol [1398] and Helindon [1460] types on wool has also been described. Bird and Newsome [1405] have reviewed the application of logwood to wool.

Carpenter [1411] has described the use of ammonium sulfate and ammonium hydroxide for buffering wool dye baths to improve leveling, and Mayants [1463] has suggested the use of a high-molecular-weight alkyl aryl polyethylene oxide condensate as a fixing agent for wool dyes. Stevens, Whewell, and Bradley [1491] have carried out a preliminary investigation of the dyeing properties of chlorinated wool, and Kramrisch [1456] has discussed the methods of dyeing wool and fur felt.

(*d*) Continuous Dyeing: Fargher [1425] has reviewed the chemical and engineering aspects of the various continuous-dyeing processes that have been proposed. He has briefly treated the Williams method, the DuPont and I. G. Farben steam processes, the Standfast molten-metal process, and others. The Standfast process has been discussed in detail by Boardman [1406], who has also outlined the difficulties which must be eliminated or minimized

in any continuous process. Brewster [1408] has described this molten-metal process more briefly. The continuous dyeing of vat colors on cotton piece goods with the Williams unit has been described [1395], and Alter [1393] has suggested an arrangement of the pad-steam process for continuously dyeing synthetics. Robinson and Zimmerman [1479] have investigated some practical aspects of the Uxbridge high-temperature dyeing machine, and Rostovtsev and Plaksin [1481] have proposed a continuous method for dyeing cotton fibers with sulfur colors.

(*e*) Printing: Peake [1475] and Fordemwalt and Witschonke [1430] have treated some general aspects of machine printing. Fordemwalt and Witschonke have been particularly concerned with the development of a method for evaluating the results in this operation.

Zonnenberg [1505] has investigated several aspects of thickening agents, especially starch, as used in printing. The effect of mechanical stirring on thickening power, the mutual influence of thickeners, color yield, and leveling power were among the factors discussed. Haller [1437] has compared the film-forming power of some of the common vegetable thickening agents, and the use of the latter in screen and hand block printing has been described [1400]. Mackenzie [1461], in discussing fine-line reproduction in screen printing, has stressed the importance of choosing the proper thickener. Two German papers [1450, 1500] have reviewed the properties of gum tragacanth and compared it with other thickeners for use in printing pastes. Schmidt [1485] has described the properties of sodium alginate and the advantages it offers in thickening printing pastes.

Berthold [1403] has suggested that the color intensification of vat prints produced by the addition of compounds such as urea may be due to the formation of organic inclusion compounds which have particles of more nearly optimum size for maximum color yield. A number of papers have discussed the use of various specific types of dyestuffs in printing —Suprafix vats [1404], Brentogens [1496], Immedialdruck dyes [1422], Anthrasols [1457], and pigments [1421, 1438].

Barth [1401] has considered the factors which are important in the steaming of prints on cellulose fabrics, and Rostovtsev [1480] has proposed a method of fixing direct dyes on cotton prints with copper or an organic fixing agent. Schramek [1486] and Hoton [1443] have reviewed the problems in printing cellulose fabrics with a number of dyestuffs. Saville [1483] has described the printing of nylon piece goods, and Thomas [1495] has discussed the screen printing of Fiberglas fabrics.

(*f*) Bleaching: The development and application of fluorescent agents for bleaching fabrics and brightening colors continues to be a very active field. Taylor [1493] has discussed the differences between fluorescence, luminescence, and phosphorescence, and the applications of each of these to textile and nontextile problems. The structure and properties of optical bleaches and the methods and conditions most suited to their use have been outlined in a number of papers [1397, 1413, 1423, 1454, 1494]. Their particular application to wool is described by Moncrieff [1468].

Hagen and an A.A.T.C.C. subcommittee have presented a collection of literature abstracts which compares hypochlorite and peroxide in continuous and semicontinuous bleaching in Part I [1434] and covers general considerations and miscellaneous processes in Part II [1435]. Klein [1453] has reviewed the behavior of cellulose during bleaching, and Kettering [1451] has made an analytical comparison of a number of bleaching methods on cotton cloth. Practical aspects of cotton bleaching have been considered by Bretcher [1407] and Fitton [1429]. Seyb and Foster [1487] have proposed a continuous hypochlorite process for bleaching cotton, and Stuhl [1492] has investigated the familiar pink discoloration which aniline produces on bleached cotton. The use of chlorites, which seem to be particularly useful with cellulosic fibers, has been discussed by De Smet [1418] and Meybeck [1467]. Pinte and Pierret [1477] and Coke [1417] have considered some of the factors in rayon bleaching, and problems in wool bleaching have been reviewed by Drake [1420].

(*g*) Stripping: Tyler [1498] has reviewed the general methods and reagents for stripping various classes of dyestuffs, and Starkie [1489] has considered the use of hydrosulfite compounds. The "Harristrip" process for stripping wool, which has been described in several papers [1399, 1424, 1478], protects the alkali-sensitive disulfide linkages in the fiber from damage during the process by stabilizing them with ethylene dibromide, which forms new cross-links with the reduced bonds. Ono [1474] has described the use of Leucosols for stripping indigoid

dyes, and Peurifoy [1476] has given some practical suggestions for the stripping of nylon hosiery.

2. Finishing.—

(*a*) General: Textile-finishing machinery manufacturers have given the textile industry its greatest number of new machines in 1950 over any similar period in its entire history [1514]. Whewell [1592, 1593] and Rinoldi [1575] have reviewed much of the work of the year that has been done on wool finishing and on the finishing of mixed fabrics of wool and viscose. Valko [1585] and Seyb [1576] have contributed systematic classifications of the chemistry of textile finishing methods and the chemicals involved. The use and application of such chemicals have been outlined by Hall [1545] and Borghetty [1518]. Wagner [1588] has described some new phosphate products and their use in finishing, while cationic softeners have been discussed by Endler and Gruenwald [1531]. Hall [1544] and Gogate [1542] have studied the mercerizing process. Shah [1578] has made some investigations of weight and dimensional changes in kier-boiling, bleaching, and finishing. A review of chemical finishes for cotton has been presented by Heberlein [1550], while the use of vinyl chloride polymers for finishing has been discussed by Kainer [1555].

(*b*) Dimensional Stabilization and Crease-Resistance: The reported developments in the field of dimensional stability have been few in number during 1950, but there has been a concerted attempt toward a better understanding of the problems inherent in dimensional stabilization and in shrinkage control. Horigan and Sage [1552] have prepared an extensive patent and literature survey on shrinkage control on textiles. Trotman [1584], Dreby [1530], and Alexander [1507] have reviewed the various processes that render wool unshrinkable, while Wengraf [1591] has considered the shrinkproofing of both cellulosic and animal fibers. An instrument which yields reproducible values for the with- and anti-scale surface friction of wool fibers in air, water, or any suitable medium has been developed by Harris Research Laboratories, whose report [1513] discusses the relationships between the differential frictional properties of the fibers and their ability to felt. Hall [1546] has reviewed several patents in the fields of shrinkproofing and has discussed the use of non-felting wool in wool and rayon-mixture materials. The chemistry of wool in relation to shrink-processing has been discussed by Alexander [1506], while Borghetty [1517] has given a general summary of the chemical and physical modifications of textile fibers as related to shrinkproofing, mothproofing, and other special finishes. Von Bergen and Clutz [1587] have discussed the dimensional changes of wool fabrics resulting from changes in relative humidity, particular emphasis being given to the role of relative humidity during tailoring. Most non-shrink treatments of wool tend to produce damage of one type or another. Various chemical and physical tests used in the determination of extent of damage have been outlined by Moncrieff [1564]. A new method of applying melamine resins to wool has been said to eliminate the need for high-temperature treatment [1536]. Whewell [1594] has given an account of the conclusions drawn from a large number of finishing trials on blended fabrics.

Woodruff has reviewed the treatment of viscose rayon fabrics to obtain shrinkage control [1595, 1596]. Compressive shrinkage of cotton fabrics has been given attention by Seydel [1577] and Pfeffer [1573]. Fletcher [1537] has determined the effect of laundering on dimensional stability and on the elastic properties of plain- and rib-knit cotton fabrics. Dietz [1528] has outlined the critical factors in the production of fabric up to finishing in so far as they relate to the ultimate shrinkage of the fabric in dyeing and finishing. A machine developed for controlling the shrinkage of cellulose acetate tricot has been reported by Walmsley [1589]. Use of a modified stenter in the successful dry-heat setting of nylon has been reported [1508]. Mecklenburgh [1561] has given a comprehensive report of the setting of nylon fabrics.

Gagliardi and coworkers [1539, 1540] have investigated the changes in physical and mechanical properties which are necessary to improve crease-resistance in cellulosic fibers. Korzen [1556] has recorded crease-resistance values for about 200 qualities of dress material, and Fischer [1535] has reviewed the use of urea and melamine resins in producing this property in fabrics. The application of various resins and other materials to wool and cotton, particularly with respect to crushproofing, has been discussed by Muus [1565] and Weiss [1590]. Anti-crease treatments for cotton, using melamine-formaldehyde precondensates, have been studied by Nickerson [1566]. The identification and prevention of the unpleasant odor which sometimes develops on resin-treated goods has been discussed in a number of papers [1538, 1557, 1581, 1582]. Another diffi-

culty encountered on resin-bearing fabrics—chlorine retention—has been investigated by Nuessle and Bernard [1567]. A new approach to producing the cross-linking which is apparently necessary for dimensional stability and crease-resistance has been indicated by Harms [1548] in a paper which has only recently become available in this country. The stiffening of fabrics, through the use of the acid colloids of some of the resins ordinarily used to produce crease-resistance, has been investigated by Nute *et al.* [1568].

(*c*) Flame-Resistance: It is now possible to treat cotton fabrics so that they are rendered more or less permanently flame-resistant. Durable methods are still expensive, however, and difficult to apply. The impetus for research on the development of fire-resistant textiles appears to stem from the textile industry itself rather than from public demand. Buck [1523] has outlined the present statutory requirements for flame-resistance in textiles. Lack of a flammability tester that will yield consistent results [1522] has contributed to confusion in this field. Johnstone [1554] and Church, Little, and Coppick [1524] have studied the evaluation of flame-resistance. Church and coworkers [1558], Heard [1549], Van Patten [1586], and Esselen [1532] have reviewed fire-retardant agents in the textile industry. Barnard [1515] has discussed the properties of flame-retardant textiles with particular reference to dermatitis, tensile strength, drape and hand, tearing-resistance, and comfort. Tests by Church and coworkers [1525] have shown that cellulose fabrics which are adequately flameproof and glow-retardant have thermal-protection qualities equal to fabrics made from the most inert fibers. Parks, Erhardt, and Roberts [1571] have investigated the catalytic decomposition of cellulosic materials and the effect of various possible flame-retardant reagents on the reactions involved.

Fain [1533] has described investigations of a number of processes for producing flameproof fabrics, and the use of titanium-antimony complexes has been considered in a number of papers [1543, 1553, 1570]. Other finishes have also been described [1510, 1511].

(*d*) Waterproofing: A literature survey on waterproofing and water-repellency of textiles for the period 1937 to 1947 has been made by Horigan and Sage [1551]. A water-impermeable, water-vapor-permeable coated fabric has been developed by Martin, Sell, and Habeck [1560]. Certain zirconium compounds [1516] when used alone render textiles somewhat water-repellent, and when used in conjunction with soaps and wax emulsions give a high degree of water-repellency. Silicone products impart water-repellent finishes to fabrics [1527] and are relatively easy to apply, particularly to nylon [1526]. Speer and Carmody [1580] have found that the titanium analogs of the alkyl silicates are capable of imparting water-repellency to most types of textile fibers. The use of paraffin and ceresin as water-repellents has been investigated by van Overbeke and Henno [1569].

(*e*) Tire Cords, Plastic Laminates, and Coated Fabrics: Hammond and Moakes [1547] have made an investigation of the adhesion of synthetic rubbers to fabrics. The technique used in measuring rubber-to-fabric adhesion [1520] and the factors which influence the load to strip rubber from fabric and foil surfaces [1519] have been studied by Borroff and Wake. The latter have also considered [1521] the adhesion of fabrics made from spun-staple yarn, particularly the function of the fiber ends, which has also been investigated by Lyons [1559]. Pittman and Thornley [1574] have proposed an improved dynamic compression test for tire-cord adhesion. The use of polyisocyanates [1562] and resorcinol [1579] for improving the bonding of rubber to textiles has been described.

The properties of high-tenacity rayon which make it useful in tires, belts, and plastic laminates have been outlined by Finlayson and Jackson [1534]. Polyester laminates of 4 commercial resins and 20 types of fabrics have been evaluated by Gallagher, Goslen, and Seymour [1541]. The interlaminar strength of such products has been studied by Pepper and Riddell [1572].

The application of rubber to wool to produce shrink-resistance [1563] and the textile uses of polyamide suspensoids [1583] and synthetic waxes [1529] have been discussed. Descriptions have appeared of a rubberized fabric which is claimed to have good properties below $-85°F$ [1512] and of the manufacture of fabrics coated with polyvinyl chloride [1509].

C. *Miscellaneous*

1. Drying.—Friedman [1602] has reviewed recent progress in general drying operations, while Ullrich [1608], Slade [1607], Bunch [1598], and Dirks [1600] have discussed various aspects of this

process as it is carried out in the textile industry. Equations for the heat required to dry textiles under various conditions of air circulation have been presented by Netz [1605]. A number of German papers have considered the application to textile drying of infrared radiation [1597, 1604, 1606] and high-frequency heating [1599]. Franz [1601] has discussed the various methods of drying wool and wool fabrics, and Klingenberg [1603], sized warps.

2. Industrial Waste Treatment and Disposal.—The pressure from public agencies for the abatement of stream pollution has been growing in recent years, and the particular problems of the textile industry have been considered in a number of papers. Hazeldine [1614], Klein [1615], and McCarthy [1616] have discussed the general aspects of textile effluents and their treatment before discharge into streams. Cotton dyeing and finishing wastes have been studied by Bogren [1610] and Coburn [1612], while Cameron [1611], Goldthorpe *et al.* [1613], and others [1248] have described some of the severe difficulties which have arisen with the increasing use of synthetic detergents in the scouring of grease wool. Balasundaram and Subrahmanyan [1609] have investigated the treatment of textile wastes so that they may be utilized as fertilizers.

3. Fiber Reclamation.—The production of reprocessed wool, which in some years has amounted to as much as 40% of the world's clip of virgin wool, is meeting with more and more difficulty with the various synthetic fibers which are being used in wool blends. In the Spring of 1950, The Textile Institute (Manchester) held a conference at Morley on the problems facing this industry. Ward [1622], Hardy [1620], and Gee [1619] have reviewed the trade and its current position, while Speakman [1621] has discussed the present reclamation methods and some that might be of future use. In this country, Dockray, Meadows, and Smith [1618] have comprehensively reviewed the cotton-waste industry, and a new continuous opener for nylon waste has been described [1617].

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J. Deterioration of Materials

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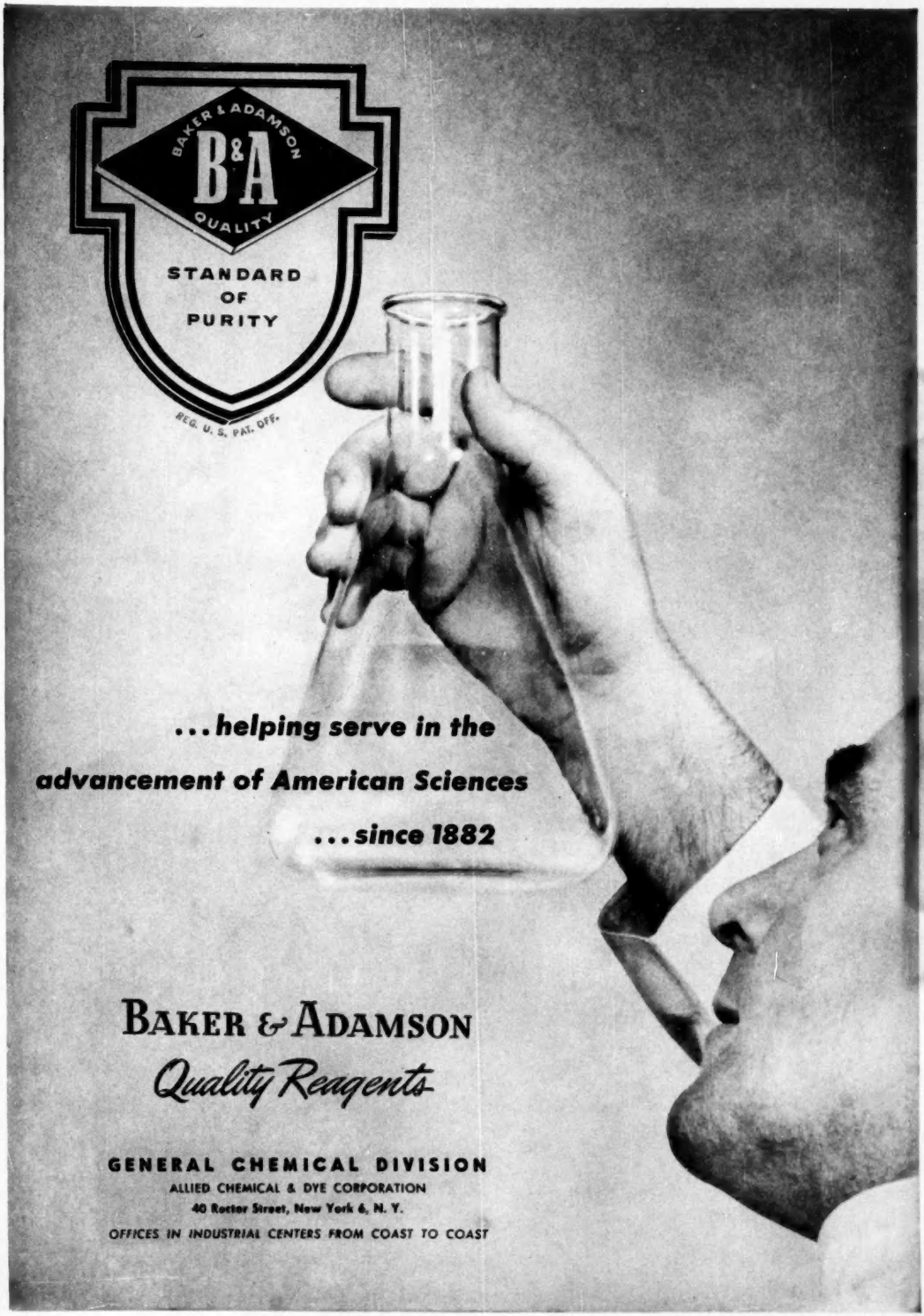
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